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VARIABLE TRANSMITTANCE
VISOR DEVELOPMENT PROGRAM (U)

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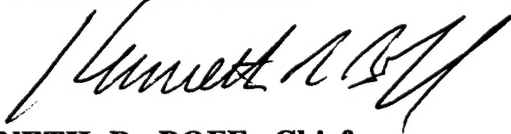AL/CF-SR-1995-0003

This report has been reviewed by the Office of Public Affairs (PA) and is releasable to the National Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

FOR THE COMMANDER

KENNETH R. BOFF, Chief
Human Engineering Division
Armstrong Laboratory

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13. ABSTRACT (Maximum 200 words)

This project's objective was to develop a technology for varying the transmittance of conventional flight-helmet visors under electronic control for the purpose of enhancing the contrast of helmet-mounted displays. The specifications required a 10:1 attenuation range with a clear-state transmittance of 70% or better and no visible coloration, haze, or optical distortion. Two technologies were studied: polymer-dispersed liquid crystal (PDLC) and suspended particles. Several low-birefringence PDLCs with neutral dye in a guest-host arrangement were tested; they produced excessive haze, apparently because their birefringence, although relatively low, was still too high for the project's purpose. The suspended-particle technology was tested in two forms: fluid and polymer-dispersed. Both forms showed a strong blue coloration in the dark state and neither proved to be compatible with conventional visors; instead, they must be sandwiched between mating halves of custom-built visors. Furthermore, over time, the fluid form developed streaks and dark spots; the polymer-dispersed form developed crazing lines and spatial non-uniformities. We conclude that the suspended-particle technology is not promising, but guest-host PDLC deserves further exploration if PDLCs having lower birefringence become available.

14. SUBJECT TERMS

Visor, liquid crystal, PDLC, suspended particle. variable transmittance, HMD, Helmet-mounted display

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FINAL REPORT

1.0 INTRODUCTION

1.1 Project Objective

A major problem associated with helmet-mounted displays (HMDs) is that the luminance of the outside scene, on which the HMD image must be superimposed, is often high enough to seriously degrade the contrast, and hence visibility, of the image. Loss of HMD image contrast can be diminished by tinting the visor to reduce the apparent luminance of the outside scene. However, if the outside scene luminance is too low, then the additional reduction in luminance caused by the tinted visor will degrade the pilot's vision. A better solution is to vary the transmittance of the visor in an inverse relationship with the outside scene luminance. Increasing scene luminance would result in decreasing visor transmittance, and conversely. A visor having this capability would also represent a very useful improvement over conventional sun visors, even if no HMD was being used.

The objective of the VTV project has been to develop a technology for varying the transmittance of helmet visors under electronic control. Such a technology would permit the above solution to the problem of the loss of HMD image contrast to be implemented in a practical system. This approach is preferable to that used in the familiar photochromic sunglasses, which respond slowly to changes in illumination, and do not permit the wearer to control the transmittance.

When using the VTV the pilot would set the see-through luminance to suit him/herself by adjusting the transmittance of the visor with a control knob. This luminance level would then be held constant at the value set by the pilot by means of a built-in photosensor and control circuit. These components would operate as a feedback control system to continually adjust the visor's transmittance in response to changes in the outside illumination level, as required, to maintain the set see-through luminance level.

The desired clear- and dark-state transmittances are 70% and 7% respectively, yielding a 10:1 attenuation range. This attenuation would result in an improvement in HMD image visibility that is equivalent, under high ambient-illumination conditions, to a 10X increase in HMD luminance. Furthermore, the 70% clear-state transmittance is much higher than the 15-20% that is typical of conventional sun visors, allowing better vision at lower outside light levels.

1.2 Technical Requirements

There are many technical requirements which an electronically-controlled VTV must meet to serve as a practical part of the Man-Machine interface in the cockpit environment. Of course, in addition to performance and safety factors, cost and maintainability are also important considerations. The principal requirements that were identified for this project are:

a) Variable transmittance performance: The VTV shall provide continuously variable and uniform adjustment of the optical transmittance of the entire visor surface. The range of attenuation shall be as high as possible, but shall be at least 10:1. Maximum luminous transmittance of the VTV shall be as close to 100% as possible, but shall be no less than 70% at all times. The mechanism shall have no observable hysteresis.

b) Optical quality: The VTV shall have no visible spatial nonuniformities in its luminous or spectral transmittance. It shall create no artifacts, aliases, MTF reductions, or other visual anomalies that would detract from pilot performance,

c) Spectral transmittance: The VTV shall have a spectral transmittance characterisitc which is as nearly flat as possible.

d) Visor compatibility: The physical parameters and material properties of the device shall be entirely compatible with the complex curved surfaces and materials of representative current visors. Increase in visor weight shall be minimal.

e) Transmittance control: VTV transmittance shall be controllable over its full range of operation both manually, by means of a transmittance control knob, and automatically by means of a photosensor and electronic control system. The photosensor shall be mounted with the VTV in order that it may accurately sense the scene luminance as seen by the pilot. The transmittance control knob and the electronic control system, which stabilizes the illumination at the pilot's eyes by adjusting the VTV transmittance in accordance with the photosensor signal, shall be incorporated in a Control Unit (CU) connected to the VTV by a cable. The time constant for variation of the VTV transmittance shall not exceed one second.

f) Electrical power: The electrical power supply for the VTV/CU device shall be completely autonomous, requiring no external supply. The VTV shall be capable of being powered by batteries or, if possible, by the photoelectric conversion to electricity of the light impinging on the visor itself. Power requirements shall be small enough to permit the operating period under battery power to be not less than four hours, but preferably as long as is practically possible. Back-up power shall be provided by a separate battery, if necessary, to insure that a failure will leave the VTV in the maximum-transmittance mode for at least fifteen minutes. The backup battery shall be helmet-mounted to prevent interruption of backup power in the event of an accidental disconnection of the VTV from the CU.

g) Environment: The device shall maintain full functionality over the complete span of normal military environmental conditions applicable to aircraft cockpits.

h) Safety: The support functions (mechanical or electronic) for the VTV shall in no way impede entry or exit of the pilot from the air-vehicle, nor present any other hazard. The device shall possess no failure modes or performance degradations that would place the pilot at hazard nor prevent the dispatch of, or in any way detract from the continued safe operation of the air-vehicle. Disconnection of support functions shall render the VTV into a "fail-safe" mode. This shall include the provision that the visor, when unintentionally disconnected from its control unit, shall revert to its maximum transmittance condition.

i) Maintainability: The maintainability and supportability of the VTV shall be consistent with normal USAF practices. Its robustness shall approximate normal visor characteristics, and should wear-out items be incorporated, these shall be cheaply and easily replaceable.

1.3 Technology Selection

There are many active (light-emitting) and passive (nonlight-emitting) display technologies which might be considered as possible candidates for use in a VTV[1-4]. This project was

conducted by examining the feasibility of all electrooptic technologies that might be applicable, selecting one or two which appear to be most appropriate and most promising, and developing these for use in the VTV.

1.3.1 Criteria for Selecting the Technologies

a) The first requirement in the selection of a technology suitable for use in a VTV is that devices utilizing it must operate as light shutters. A light shutter controls the amount of light which can pass through from an outside source, rather than generating light directly. Because the purpose of a VTV is to control the transmission of light from the environment to the pilot's eyes, a light shutter is required, and light emitting technologies are therefore ruled out. Therefore, light-emitting technologies, such as CRT's, electroluminescent (EL) panels, and LEDs are inappropriate for this application because none of these function as light shutters.

b) Only those passive light-modulating shutter technologies which have good contrast ratio (>10:1) and high transmittance (>50% minimum) need be investigated. Those not able to achieve these levels of contrast and transmittance will not meet the performance requirements of the VTV.

c) Light absorbing technologies are preferable to light scattering technologies. Light absorbing devices reduce the transmitted light by absorbing some fraction of the light incident upon them. An image seen through an absorbing light shutter (of good optical quality) remains clear as the image is dimmed. Light scattering devices, on the other hand, reduce the amount of light passing straight through the shutter by scattering it away in other directions. An image seen through this type of shutter appears hazy as it is dimmed. Some types of light shutters may exhibit a combination of both absorbing and scattering behaviour. Such devices might be useful in a VTV provided the scattering component is sufficiently small.

d) Shutter technologies capable of producing devices in flexible sheet form compatible with the curved surface of the visor are preferable. A flexible light shutter could be wrapped around the curved surface of a visor and laminated onto it, although this is likely to be difficult on surfaces curved in two axes. If a laminating approach is feasible, this would make the production of VTVs easier and cheaper than it would be if rigid curved shutters had to be made. One reason for the lower cost is that standard visors could probably be used. Such VTVs would also be lighter in weight than those made using rigid light shutters. The weight would not be significantly greater than that of the visor itself.

1.3.2 Possible Candidate Technologies

Based on the above criteria, candidate shutter technologies for the VTV have been selected. Those which appear to be the most feasible include suspended particle, liquid crystal, electrochromic, and electrophoretic technologies. Each of these, of course, has its own set of limitations, advantages, and disadvantages with respect to the VTV application. Some preliminary studies and measurements were also performed to test the suitability of these technologies for use in the VTV.

1.3.2.1 Liquid Crystal Shutters

Liquid crystal (LC) displays and shutters are passive devices which exist in a variety of forms. These include TN mode, dynamic scattering, supertwist, ferroelectric, Heilmeier, phase

change, microencapsulated, active-matrix, etc[2]. Supertwist and ferroelectric have very sharp threshold characteristics (i.e., contrast vs voltage response) and are therefore not very suitable for a continuously dimmable visor. Moreover, they also require a very thin cell gap and very precise thickness control, which will be difficult to maintain in curved cells. Although the required cell gap is larger in other types of LC devices, which relaxes the thickness control problems somewhat, cell gap uniformity is still critical. Therefore, the curved shape of the visor poses difficult problems for most liquid crystal shutters.

With these reservations in mind, several types of liquid crystal shutters were considered for the VTV. These are TN, dynamic scattering, Polymer Dispersed, and dye doped Polymer Dispersed.

TN Liquid Crystal[5]: TN LC shutters have the advantage of being an established technology. TN LCDs are very well known in consumer products such as watches and calculators. They are extremely good light modulating devices, capable of high contrast (>50:1), they are light absorbing rather than light scattering, they operate at low voltages (5 volts), and have low power consumption. Moreover, they can be made in clear as well as dark mode. Clear mode shutters become dark, and dark mode shutters become clear when voltage is applied. Clear mode shutters are, of course, inherently more fail-safe. However, the transmittance of TN LC shutters is low (35%) and, in their conventional glass-cell form, are not very suitable for curved surfaces. They also display a viewing angle dependence, although this would be partially compensated by the curved shape of the visor.

TN LC shutters using polarizers as substrates might have some application for VTVs. It is possible that the glass substrates could be replaced by the polarizers themselves, with the conductive Indium Tin Oxide (ITO) coatings being applied to the polarizers directly rather than to glass substrates. Such a shutter might be flexible enough to take the shape of the visor.

Dynamic scattering LC[6]: Dynamic scattering LC shutters have high transmittance (>70%), high contrast (>10:1), operate at fairly low voltage (15-20V), and require low power ($\mu W/cm^2$). On the other hand, they operate by light scattering rather than by light absorption. The shutters are clear in the unactivated off-state, but on application of voltage, scattering centres are created and they become milky white. Dynamic scattering mode devices also have a shorter life than other LC devies. In addition, they are not very flexible, normally being made with glass substrates. It might be feasible, however, to make such shutters with plastic substrates.

Polymer Dispersed LC[7]: Normal scattering mode Polymer Dispersed (or microencapsulated) LC shutters consist of two transparent electrically conducting ITO-coated plastic films enclosing a thin layer of microencapsulated nematic liquid crystal droplets in a polymer matrix. There are two versions of microencapsulated LCDs, depending on the method used to form the droplets. In both types, however, the shutter scatters the light strongly in the quiescent condition. When the electric field is applied, the liquid crystal molecules in the droplets become aligned in the direction of the field and the shutter becomes clear. These devices show a very good grey-scale characteristic, which is a desireable property for the VTV.

These shutters operate in the 40-60 V range, have good contrast (>10:1), good transmittance (>60%), operate at low power, and can be made in the form of a thin, flexible plastic sheet. Since the microscopic droplets of liquid crystal are enclosed in a polymer matrix, these shutters have high hermeticity, and consequently high-reliability, environmental stability and long life. They can also be operated over a wide temperature range. No polarizer is required.

It is possible that one surface of the visor could be coated with ITO to serve as one electrode of the shutter. The liquid crystal material would then be applied directly to the coated

visor, and a second layer of ITO would be applied on top. In this way the shutter would become an integral part of the visor.

The primary disadvantage of the Polymer Dispersed LC shutters is the scattering, although there is some possibility that a haze-free mode might be feasible.

Dye Doped Polymer Dispersed LC: Pleochroic dyes can also be added to liquid crystal to make another version of the microencapsulated shutter. The dye makes the device absorbing in the quiescent state, so that the shutter becomes dark in the off-state. Upon application of the electric field, the dyes, along with the liquid crystal, align in the direction of the field, and the shutter becomes clear. The operation of these devices, then depends much more strongly on absorption of light rather than scattering. Dye Doped Polymer Dispersed LC shutters have good transmittances (~50%), good contrast (>10:1), and can be made in flexible sheet form.

This type of shutter appears to be a very strong candidate for the VTV application. However, not much work has been done on this technology to date, and it is therefore still in the beginning stage of its development.

1.3.2.2 Electrochromic and Electrophoretic Shutters[8]

An electrochromic material changes its colour by application of an electric field. There are two principal versions, one using inorganic WO_3 thin-films, and the other using organic materials such as violagens.

Electrophoretic devices exploit the phenomenon of electrophoresis, which is based on the motion of charged particles in a fluid under the action of an applied electric field. The charged pigment particles, typically 1 μm in diameter, are suspended in the fluid by chemical techniques. On application of the electric field, the particles are attracted to, and deposit on the electrodes.

Although electrochromic and electrophoretic devices are both highly transmissive with good contrast, and operate at low voltages, they both consume more power than liquid crystal devices, their life is unknown, and they are made using hard glass substrates. Neither technology has been perfected, and they are presently little more than laboratory curiosities. Because it is believed that a great deal of development is still necessary before these technologies would be practical for use in a VTV, they are not considered good candidates for the VTV at this time.

1.3.2.3 Suspended Particle Shutters[9-11]

Suspended Particle technology is based on the use of light polarising and light absorbing particles suspended in a liquid medium. The particles are oriented by an electric field to turn the shutter on, and disoriented by Brownian motion to turn the shutter off. Suspended particle shutters are primarily light aborbing rather than light scattering. They operate at moderate voltages (50-60 volts, or less), have fairly low power consumption (mW/cm² at low operating frequencies), have good contrast (>10:1), and have good transmittance (60% or higher). They are relatively easy to fabricate, and there is the future possibility of making them in microencapsulated sheet form. Suspended particle technology appears to be a very strong candidate for the VTV application.

1.3.3 Most Appropriate Technologies for VTV Applications

Consideration of the above possible technologies and their characteristics, which are pertinent to the timely development of a working VTV, led to the selection of the most likely can-

didates. The candidates chosen were suspended particle shutters, of either liquid type or film type, and dichroic Polymer Dispersed Liquid Crystal (dichroic PDLC). Hence, these are the technologies which were investigated over the duration of this project. Of the two technologies, it was thought that suspended particle technology showed the greater promise in the short term, and more effort was therefore expended in this area.

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2.0 SUSPENDED PARTICLE SHUTTER TECHNOLOGY

2.1 Introduction

The principal technology which was investigated for the VTV application is suspended particle (SP) technology.[1,2,3] The SP material is essentially a fluid containing a colloidal suspension of microscopic anisometrically-shaped solid particles, consisting of needle or plate-shaped crystals. The particles are light polarizing and absorbing, with a length of about 1 μm or less. The suspension is stabilized against agglomeration by means of special polymers surrounding the crystals. It is necessary to use a polymer when forming the crystals in order to prevent overgrowth of the crystals, and also to minimize the agglomeration of crystals together into clumps. In the final suspension, these polymers continue to isolate the crystals from each other, while keeping them suspended in the fluid. The specific gravity of the suspension is also balanced with respect to the crystals and polymer to prevent gravitational settling of the particles.

SP light shutters (or cells) consist of a thin layer of suspended particle material sandwiched between indium-tin oxide (ITO) coated glass or plastic substrates, with an epoxy or thermoplastic peripheral seal.

When a cell is unactivated the suspended particles remain disoriented, or randomly aligned by Brownian motion. In this state the particles have a large absorption cross-section, as well as a small scattering cross section, and block the incident light. When the cell is activated by applying a voltage between the ITO layers in the cell, the particles aquire an induced dipole moment. The aligning torques produced by the electric field then override the random torques produced by Brownian motion, and the cell becomes essentially transparent to the incident light. Hence, SP shutters are normally dark in the unpowered state, and transparent in the powered state. Switching speed is also nonsymmetric. Turn-on is always faster than turn-off because turn-on is driven by the applied electric field, whereas turn-off relies on the passive action of the Brownian motion. Operation of a SP cell is illustrated in Figure 2-1.

Two basic types of suspended particle materials were investigated for use in a VTV; fluid suspensions, and films of silicone-based polymers in which tiny capsules of fluid suspension are dispersed. The latter materials may be referred to as Polymer Dispersed Suspended Particle (PDSP) materials, analagous to Polymer Dispersed Liquid Crystal (PDLC) or Nematic Curvilinear Aligned Phases (NCAP) materials. In each type of SP material the operating principle is essentially the same, although the physical properties of the materials are quite different. Both types of SP materials appear blue in colour, particularly in cells that are dark. This colour is characteristic of the crystals, and is a result of their iodine content. These materials, and the work done with them for the VTV application are described in the remainder of the report.

The suspended particle crystal materials used for research and development purposes at LSL were supplied by Research Frontiers Inc. (RFI), Woodbury NY. Most of these materials were produced at RFI as experimental materials, and were undergoing continual development throughout the duration of the VTV program. SP fluids were generally supplied in directly useable form, whereas all SP films were prepared at LSL using the component chemicals supplied by RFI. In some cases, generic chemicals, which were available commercially, were purchased from chemical suppliers.

LIGHT VALVE "OFF"

LIGHT VALVE "ON"

INCIDENT LIGHT BEAM

INCIDENT LIGHT BEAM

TRANSMITTED LIGHT BEAM

- NO FIELD APPLIED
- PARTICLES RANDOMLY ORIENTED BY BROWNIAN MOTION
- LIGHT SCATTERED AND ABSORBED

- ELECTRIC FIELD APPLIED
- PARTICLES ELECTRICALLY POLARIZED
- PARTICLES ALIGNED PARALLEL TO FIELD
- LIGHT TRANSMITTED
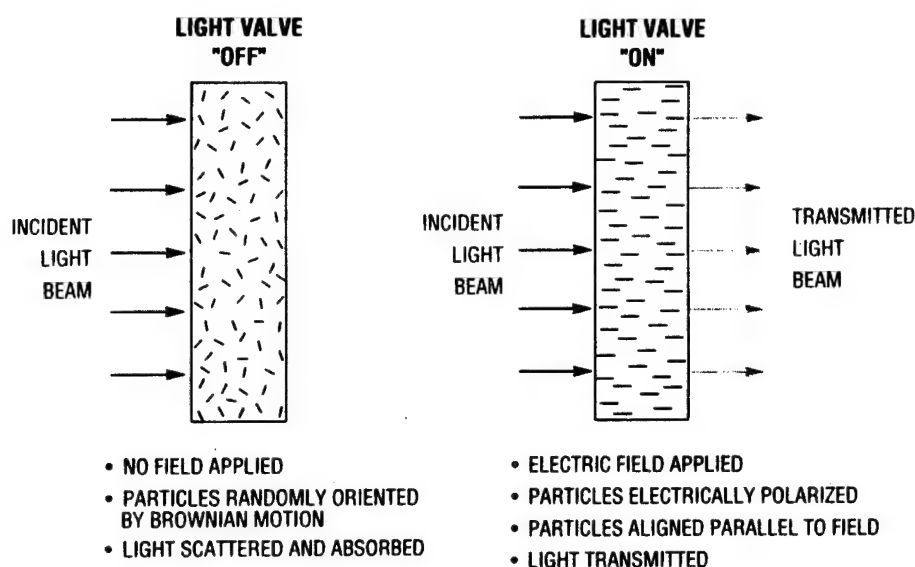
Figure 2-1. Suspended Particle light shutter.

2.2 Fluid Type Suspended Particle Cells

2.2.1 SP Fluids

The first type of SP material available was the fluid type suspension,[4,5] and all of the early work at LSL was conducted using many different variations of this material. The first type of fluid investigated at LSL contained crystals of dihydrocinchonidine sulfate periodide suspended in a mixture of halocarbon oil and an ester such as neopentyl neopentanoate, dioctyl adipate or diisodecyl adipate or a trimellitate such as tri-n-propyl trimellitate, and using nitro-cellulose and neopentyl acrylate/methylol acrylamide copolymer as suspending polymers. Many variations, however, are possible. The mixture of liquids in any particular suspension is adjusted to achieve a density that will gravitationally balance the particles and associated polymer(s). The proportion of crystals in the suspension may also be varied in order to change the optical density of the cells, or to compensate for different cell thicknesses. The suspensions were mixed at RFI to the particle densities required to match the test cells being used at LSL.

Further developments at RFI led to improved suspensions containing crystals of 2,5-pyr-azinedicarboxylic acid polyiodide as the suspended particles, nitrocellulose and neopentyl meth-acrylate/hyroxyethyl methacrylate copolymer as the suspending polymers, and a mixture of tri-n-propyl trimellitate and halocarbon oil as the suspending liquid (or solvent). This type of suspension promised to be more stable than the earlier ones with respect to agglomeration, set-tling, and exposure to temperatures above 85° C, and to give better electooptical performance.

The molecular structure and molecular weight of the suspending polymer or polymers has a very significant effect on the properties and performance of the suspension. Factors such as cell operating voltage, stability of the suspension against agglomeration of the particles into clumps or clusters, speed, and resistance to high temperatures are all affected by polymer design. For example, polymers containing branched monomers reduce the field strength needed to align the particles in the suspension, compared to unbranched monomers. Hence, cells can be

made to operate at lower voltages using such materials. Polymers containing branched monomers also tend to reduce the prevalence of crystal agglomeration, probably because the solubility of such polymers in the suspending fluid is higher. In addition, these polymers make the suspension more resistant to raised temperatures. Molecular weight also affects agglomeration, as well as speed. Higher molecular weight improves stability, but also generally increases viscosity, and hence decreases operating speed. Therefore, molecular weight must be chosen to provide a satisfactory trade-off between these opposing requirements. This, of course, assumes that polymers of a desired molecular weight and structure can be produced, and that the molecular weight will be stable with time without further polymerization taking place. In practice, this has turned out to be difficult to achieve.

Development of more effective suspending polymers constituted a major area of research at RFI throughout this program, and most of the promise of improvements in performance were based on projected improvements in the suspending polymers. Problems with the polymers, in terms of design and synthesis, were also the basis of most of the performance problems with the suspensions.

A great deal of work was done with both of the above types of fluid suspensions at LSL. A major component of this work was the development of methods for handling and controlling fluids and filling cells in a vacuum chamber. This work is described in the following sections.

2.2.2 Cell Fabrication

Cells for fluid SP shutters consist of two ITO-coated glass or plastic sheets with a very narrow gap between. The width of the gap is typically about 10 μm to about 60 or 70 μm, or more. Thicker cells have the advantage that it is easier to maintain the accuracy and uniformity of the gap thickness, as a percentage of overall thickness. It is also thought that thicker cells might help to reduce agglomeration. On the other hand, the thicker the cell the higher the operating voltage required to operate the completed light shutter. Operating voltages are about 1 to 2 volts per micrometer of cell gap, depending on the particular suspension.

Gap thickness is maintained by the use of spacers which are spread randomly over the cell area. Spacers are glass or plastic, in the form of spheres or short pieces of fiber. Another disadvantage of thick cells is that the spacers are more noticeable than in thin cells. Clear spacers appear as bright specks when the cell is in the off-state (dark state), and black spacers appear as dark specks in the on-state. It is possible to maintain cell gap in small cells with strong walls using only a single circumferential spacer, but this would not be feasible in the VTV.

Figure 2-2 illustrates the construction of a basic suspended particle cell using a simple peripheral spacer.

Uniformity of cell gap is very important, and must be maintained to within a few percent in order that the optical density of the cell will appear uniform. Achieving good uniformity and stability of gap thickness is more difficult with VTVs than with simple flat glass cells. There are two principal reasons for this: the visors are made of plastic, which is less rigid than glass and is more subject to flexure in handling and use, and they have a non-spherical compound curve which requires that both front and rear parts must be accurately aligned with respect to each other for their two curves to properly match.

Cells for fluid-filled shutters are completely assembled prior to filling. After the substrates have been thoroughly cleaned the spacers are spread onto one of the substrates. This may

be done by allowing the spacers to settle onto the substrate in air in a special settling tower, or by spraying a suspension of spacers in a volatile liquid onto the substrate. The second substrate is then placed over the first and held in position while the periphery of the cell is sealed with epoxy. A hole is left in the seal through which the cell may be subsequently filled with SP fluid.
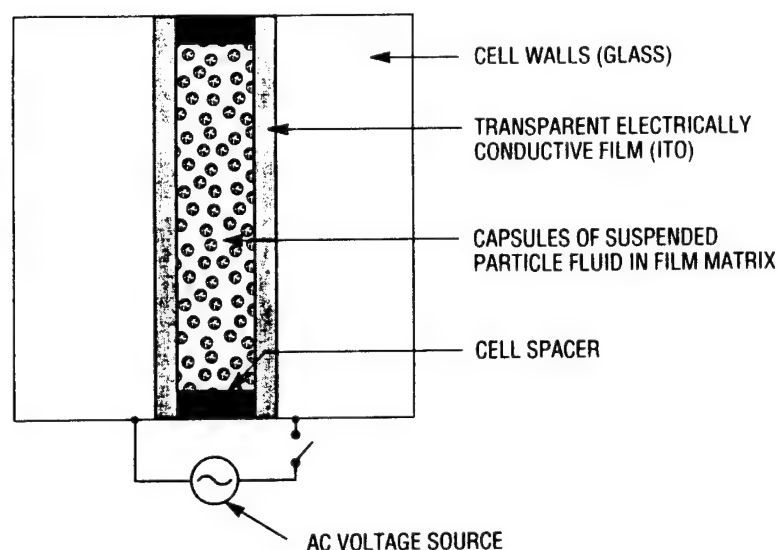
Figure 2-2. Cross section of Suspended Particle shutter.

Many test cells were fabricated and filled, in sizes from 1" x 2" up to 12" x 12", the majority being 2" x 2". These cell-making experiments were used to study the effects of varying a multitude of process parameters, and other factors on the optical quality of the cells, and to evaluate the performance of the various SP fluids obtained from RFI.

2.2.3 Cell Filling

Some of the basic process development work had been performed before the beginning of the VTV project, so that a fairly good understanding of how to fill liquid type SP cells, and of the problems remaining to be solved had been aquired. Development of these processes, and attention to the many remaining problem areas, however, continued throughout the VTV program. This work is described in the following paragraphs.

Various methods may be employed to fill SP fluid cells. If two fill holes are provided on opposite edges of the cells, rather than only one, then the cells may be filled by capillary action through one of the holes. Air then escapes through the other hole. Alternatively, a fixture may be used to allow air pressure to force the fluid into the cell through one of the holes, or a partial vacuum applied at one hole may be used to draw the fluid into the cell through the other hole. The most significant problem with these methods is that air bubbles usually become trapped in the cells, often in the corners. This can be alleviated to some degree by allowing the fluid to flow through the cells to flush bubbles out. However, this does not produce reliable results, and cannot easily be used with the capillary fill method. Two entire opposite edges of a

cell may be left open to permit a uniform flow across the cell and, hence, to avoid blind spots where bubbles may become trapped. However, this method is not considered to be practical for most cells.

At LSL SP test cells are filled using a vacuum filling technique. Cells to be filled are placed inside a vacuum chamber, which is then pumped down to less than 1 mT pressure. This pressure is maintained for at least 20 minutes to allow sufficient time for the cell internal pressure to reach the chamber pressure through the small opening of the (single) filling hole. At this pressure residual air left inside the cell, when compressed back to atmospheric pressure when the cell is filled, will yield only a microscopic bubble which will readily dissolve in the fluid and disappear. The vacuum filling method has proven to be very effective, and when done properly results in cells completely free of any bubbles. Vacuum filling of cells with SP fluid, nevertheless, is by no means free of problems. These problems, and solutions which have been developed at LSL, are discussed below.

The vacuum chamber used at LSL consists of a circular cylinder of stainless steel 17 inches in diameter and 6 inches high with machined flanges top and bottom and six access ports equally spaced around the edges. (This is a standard vacuum system component referred to as a feed-through collar.) The bottom is closed by an aluminum plate and gasket, below which is mounted a high vacuum valve and difussion pump. The top is closed by a thick transparent sheet of polycarbonate, which may be lifted off to gain access to the chamber. A mechanical roughing pump, valves, and vacuum guages essentially complete the system.

One of the problems of the vacuum fill method is that any volatile components of the SP fluid will be lost in the vacuum. As stated earlier, the fluid contains halocarbon oil, which has a vapour pressure of approximately 10 Torr at room temperature. This material will therefore evaporate quickly in the vacuum required for filling cells. This problem can be circumvented by establishing an atmosphere of halocarbon oil vapour in the chamber during fill, up to its vapour pressure at room temperature. This cannot be accomplished, however, until after the pumping cycle is complete, otherwise the vapour would be continually pumped away. Among other problems, this would result in the loss of a lot of oil, and is incompatible with the operation of the difussion pump. The use of a difussion pump is necessary to extract the air to the required low pressure.

A special system was developed to fill the chamber with oil vapour, consisting of a heated reservoir (stainless steel sample bottle) for the halocarbon oil, tubing and valves. These are all external to the vacuum chamber. The oil in the reservoir is degassed, and kept under vacuum. Before use, the oil is heated a few degrees above room temperature by an electric heater wrapped around the reservoir in order to raise its vapour pressure a little above its room temperature value. After pump-down, the high vacuum valve is closed to terminate pumping, and the vapour is admitted to the evacuated chamber by opening a valve leading to the oil reservoir. Several minutes later, halocarbon oil begins to condense on the walls of the vacuum chamber because of the slight excess of vapour pressure with respect to room temperature. This indicates that vapour saturation has been reached in the chamber, and the halocarbon oil valve is then closed.

The SP fluid, which will be used to fill the cell, cannot be introduced into the vacuum chamber until the atmosphere of halocarbon oil vapour has been established, otherwise halocarbon oil would be lost from the SP fluid. When the chamber is ready, a small reservoir surrounding the filling hole in the cell is filled with a controlled volume of fluid. The reservoir may take the form of a small trough in which the cell sits vertically with the fill hole at the bottom. The fluid is introduced into the vacuum chamber through a system of tubes and valves from an external air-tight reservoir where it is stored until needed. Air is then vented into the

chamber to raise the pressure a measured amount, and this pressure forces the fluid into the cell through the fill hole. Even without introducing air into the vacuum chamber, the cell would eventually fill due to capillary action alone. However, it has been found that the quality of the finished cell depends to some degree on the filling rate, and hence the chamber pressure during fill.

Before the fluid can be allowed into the cell, however, it must be degassed to remove¯ dissolved air. If this is not done, there is a high risk of bubbles forming in the finished cell. Ideally, the fluid may be degassed prior to use and stored under vacuum, except for an atmosphere of halocarbon oil, in the sample bottle in which it is normally kept. This method was eventually abandoned, however, because of difficulty in getting the fluid to flow from its container into the vacuum chamber. Flow will only occur under gravity because there is no pressure differential to force the fluid through the valve and tubing. This was found to work quite well when using low viscosity fluids, but not with the more common higher viscosity materials. Larger diameter tubing could have been used to solve this problem, however, this resulted in wasting too much fluid each time a cell was filled because of the volume required to fill the tubing. The quantities of fluid available for experiments were small and it was important to conserve it.

An alternative method was adopted in which un-degassed fluid at or near atmospheric pressure was introduced directly into the vacuum chamber through a fine teflon supply tube and made to flow from the end of this tube down a short length of inclined tubing of much larger diameter (about 1 cm inside diameter). This process exposed a large surface area of the fluid to the vacuum for 15 or 20 seconds before it dripped off the tapered end of the tube into the filling trough. The volume of the vacuum chamber is large enough that the small amount of evolved air had little effect on the chamber pressure prior to the cell filling hole being closed by the fluid, and it was rare for any bubbles to appear in completed cells.

When the cell is completely filled, it is removed from the vacuum chamber, the excess fluid cleaned off, and the fill hole sealed with epoxy. The expoxy is cured at room temperature to prevent thermal expansion of the fluid inside the cell, which would force the fluid out of the fill hole.

2.2.4 Fluid Test Cell Experiments

2.2.4.1 Performance Problems in Early Cells

Process development and performance evaluation experiments, were first conducted using the dihydrocinchonidine sulfate periodide type suspensions. During this period, cells covering a wide range of sizes were fabricated, which verified the basic feasibility of the vacuum fill method. Since most of the work with this material was conducted prior to the beginning of the VTV program, it will not be discussed further, except to say that a basic problem with the suspensions was first seen during this time. This problem manifested itself in the form of localized optical density variations which appeared in the test cells. These usually appeared as linear or curved streaks, or spots. In most cases these marks were darker than the surrounding material, although sometimes lighter ones were seen. In many cases, the cosmetic appearance of the cells degraded after being operated, with the appearance of more marks or density variations. Some cells also seemed to become worse with time. In some cases these structures were quite small, so that the cells looked visually fairly smooth overall, provided they were not examined closely.

It was initially assumed that these marks were caused by problems with the fill procedure, or by contamination of the cells. Considerable effort was expended trying to prevent

these marks from occuring by means such as taking greater precautions to insure cleanliness during cell assembly, and varying fill process parameters such as chamber pressure before and during fill, and fill rate. Other factors such as fill hole size, cell size, cell gap, epoxy type etc. were also examined for their possible role in this problem. However, the defects continued to appear. Some suspension formulations were better than others. Suspensions that were designed to have lower viscosity for the purpose of increasing operating speed, generally showed more agglomeration (and hence spots and streaks) than higher viscosity ones.

In discussions with RFI, various potential problems with our fill procedure were suggested, and these were investigated. However, improved suspending polymers were under development at RFI, and these were expected to greatly increase the stability of the suspensions and eliminate the streaking problem.

2.2.4.2 Experiments with New Pyrazine Suspensions

Shortly prior to the beginning of the VTV program, RFI announced to LSL that they had developed a new suspension which used a new type of crystal material, as well as new polymers. This was the 2,5-pyrazinedicarboxylic acid polyiodide type suspension mentioned in the Introduction. A program of work on this material then commenced at LSL as soon as research material became available from RFI. Subsequently, all fluid suspensions investigated at LSL were variations on this class of suspensions.

Many test cells were made with cell gaps around 25-27 μm, which operated at voltages in the 40-50 volt range. Other series of test cells having gap widths of 15 μm and 10 μm were also investigated, with the objective of reducing operating voltages required to achieve full contrast. Operating voltages as low as about 20 volts were achieved in the 10 μm cells.

In addition to the elimination of the visual defects, the principal performance improvements which were sought were higher contrast ratios, faster speeds and lower operating voltages. As already mentioned, higher speed implies lower fluid viscosity which, in turn, generally means lower molecular weight suspending polymers and lower stability of the suspension. Lower operating voltages generally imply thinner cells and, therefore, higher concentrations of crystals required in the suspensions in order to achieve the same optical densities. Higher crystal concentrations, in turn, increases the difficulty of achieving high stability. Therefore, many different fluid variations were tried to study the trade-offs involved in these conflicting requirements.

2.2.4.3 Performance of Pyrazine Type Cells

Speeds in the 100 ms range were achieved with the fastest suspensions, and contrast ratios around 10:1 with on-state transmittances of about 50% and off-state transmittances of about 5% were commonly observed, depending on the particular suspension. Contrast ratios of about 40-50, or even higher were achieved in dark cells with certain suspensions, typically with off-state transmittances around 1-1 1/2%, and on-state transmittances around 50-60%.

Figure 2-3 shows the shape of a typical threshold characteristic curve (transmittance vs voltage) for a fluid cell. The curve indicates that these cells display good grey scale characteristics, so that transmittance can be readily controlled over the full contrast range by varying the voltage.

The best performance figures for speed, contrast, and voltage could not be achieved simultaneously. Thin cells, which could operate at low voltages, could not achieve high contrast because the off-state could not be made dark enough. This resulted from a limitation in SP

technology with respect to the maximum practical concentration of crystals which could be used in the suspensions. The highest speeds were also not achieved in the highest contrast cells. Higher contrast cells tended to be slower, typically around the 1 second speed range in decay.

TRANSMISSION %

VOLTAGE
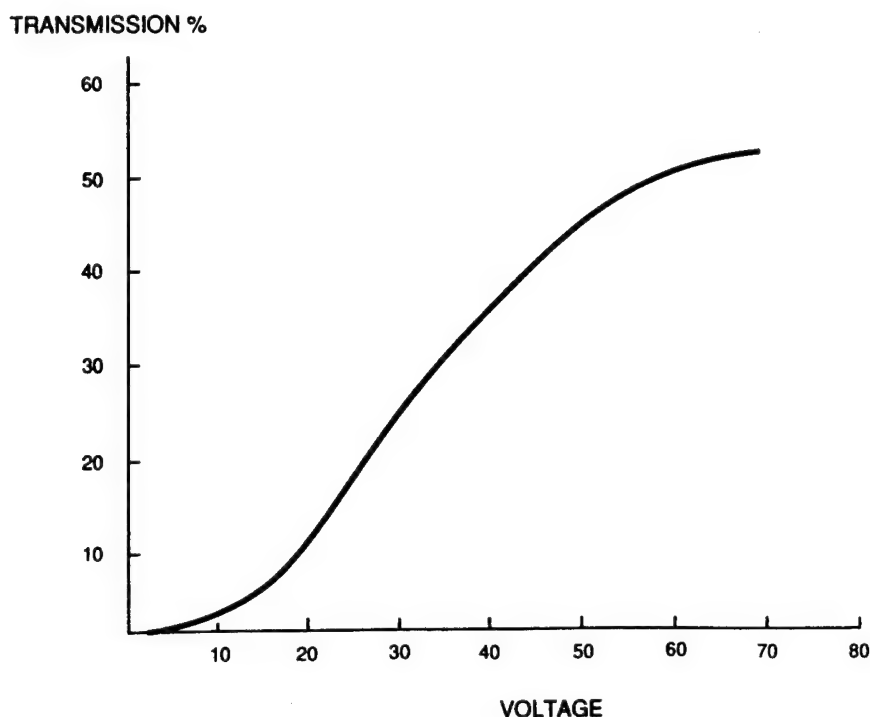
Figure 2-3. Threshold characteristic of a fluid cell for 0° inclination angle.

Figure 2-4 shows the spectral transmittances of a fluid cell in the on and off states. The rise in the transmittance toward the blue end of the spectrum in the off state, which gives the SP shutters their blue colour, can be easily seen.

Figure 2-5 shows the haze performance of a fluid cell as a function of voltage. It is clear that the haze degreases substantially as the cell turns on, and is very low at full transmittance.

2.2.4.4 Performance Problems in Pyrazine Type Cells

The problem of the formation of spots and streaks observed in earlier cells also continued. This was despite the use of new and improved suspending polymers in the pyrazine type suspensions, which were expected to improve stability. Usually these defects appeared immediately when the cells were filled. In one series of cell-filling experiments, streakiness appeared primarily in a band along the edge of the cell furthest removed from the fill hole. Often, semicircular zones of different optical densities appeared around the fill hole itself.

In some cells dark spots appeared subsequent to filling. Some of the 10 μm cells were the most uniform and best looking fluid cells ever produced at LSL, just after they were completed. In these cells no visible defects or density variations could be seen. In making these cell

the fluid was filtered through a fine glass filter just prior to filling the cells. However, within days small, dark, hazy spots began to appear at random within the cells, and these became very noticeable within a week or two. Once these marks had formed, they became permanent and did not fade away again.
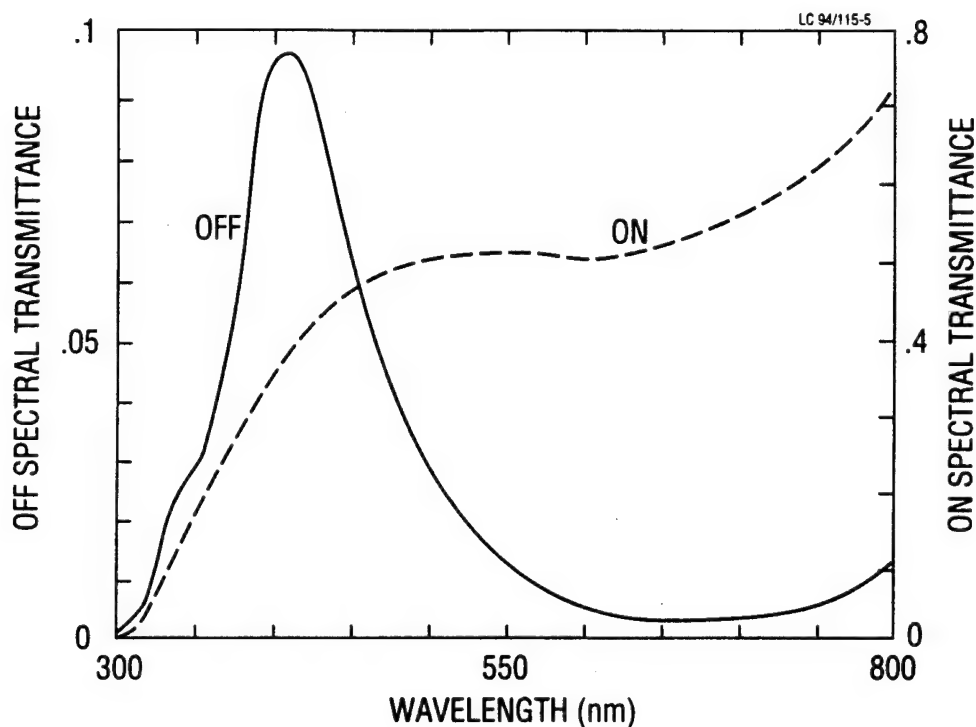
Figure 2-4. On- and Off-state spectral transmittances of a fluid cell.

Microscopic examination of the spots and streaks showed that, in a few cases, the spots were associated with stray contaminating particles inside the cells, or broken fragments of spacers. In most cases, however, no foreign particles were visible in or around the spots, and the spots and streaks simply appeared to be hazy regions where the density of crystals was slightly higher than in the surrounding suspension. It appears that something was causing the suspended particles to spontaneously agglomerate even under static conditions, and in the absense of foreign particles.

As a point of interest it should be noted that this is in contrast to some of the earlier cells made with the dihydrocinchonidine type suspensions, in which dark spots were often found to be associated with microscopic contaminating particles. Sometimes when these cells were first energized, arcing would occur within the cells, which appeared like tiny flashes of light. Usually these were short-lived, but left permanent visible marks in the cell, sometimes with concentric rings of density variation around them. Microscopic examination revealed a small irregular piece of foreign material, usually black in colour, in the center of each such area of cell damage. Presumably these were conducting particles which short-circuited the cell, or at least locally shortened the cell gap suficiently to cause a spark to jump the gap. This rarely occurs with the newer pyrazine materials, suggesting that these suspensions are cleaner

than the older ones.
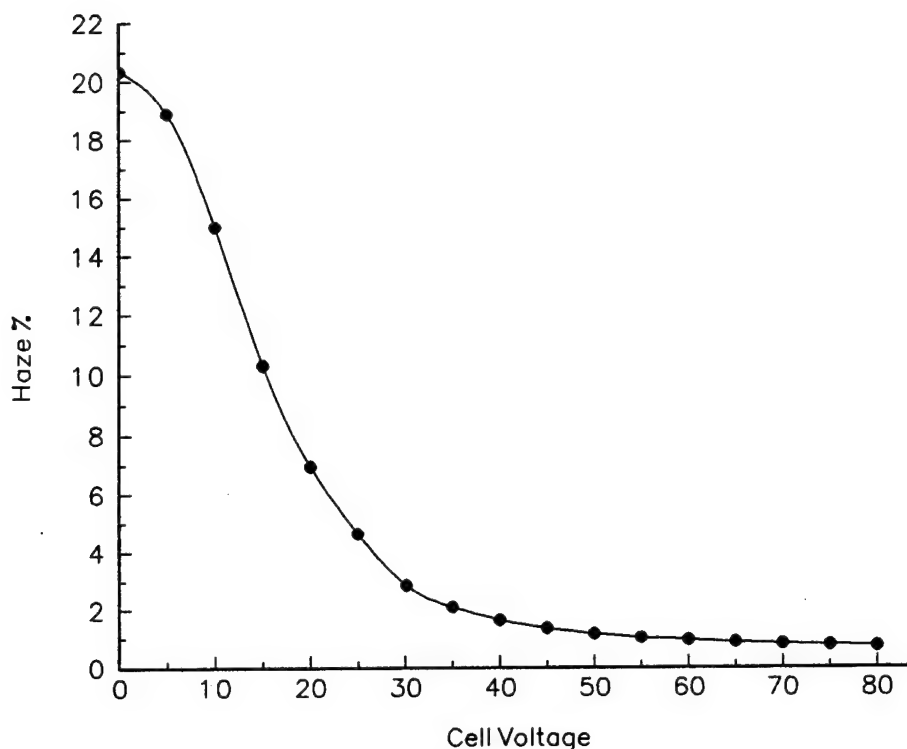
Figure 2-5. Percent haze of a fluid cell.

One would expect the SP material to be self-healing in the sense that any density varia- tions which might occur in the suspensions should diffuse away as a result of the Brownian motion. This does actually occur to some extent in those cases where the nonuniformities appear immediately upon filling the cells, although some residual nonuniformity usually remains. The process generally takes many weeks. However, spots that appear spontaneously, despite the action of the Brownian motion, do not appear to diffuse away again, as mentioned ealier. These observations imply that the suspension is not sufficiently stable against agglomeration, and that the suspending polymer system requires further improvement.

RFI recommended heating the cells in an oven to increase Brownian motion in an effort to diffuse away the irregularities. A number of trials were conducted to test this idea, with only partial success. Some smoothing of density irregularities was observed in some of the cells, but in other cases it appeared to make the irregularities more prominent.

It has been found that cells made with expanded SP films gradually lose their contrast with time, which appears to be a result of the gradual loss of the solvent through evaporation. Currently, all the early cells made in this way show little, if any, change in transmittance when activated. Possibly, the dimethyloctane diffuses through the epoxy seals around the cells. On the other hand, if the demethyloctane (or other solvent) mixes with the epoxy while it is being applied, channels can form in the epoxy through which the solvent may pass. This could

happen because the solvent normally fills the cells right to the edges, and it is somewhat difficult to keep the surfaces to be bonded dry and free of solvent while sealing the cells.

RFI reported to LSL that it observed deterioration of the ITO coating in contact with both halocarbon oil and UV-curing adhesive used to seal the cells. This led to deterioration of the cells, and even to interruption of the electrical path to the connection pads. This problem was not observed at LSL. However, the solvent used at LSL was primarily dimethyloctane, rather than halocarbon oil, and the sealing epoxy was thermal-curing rather than UV-curing. Furthermore, fluid-filled cells using halocarbon oil as the solvent, did not show this problem either.

2.2.4.5 Possible Causes of Process-Related Problems

There are many possible factors which might either cause or contribute to the agglomeration problem other than simply an inherent instability in the suspensions. Each of these was investigated, and is briefly described below.

It is believed that a kind of flow separation process occurs in the suspension as the material is forced through the narrow filling hole, encounters the sharp edges of the glass substrates at the entrance to the fill hole, or flows around obstacles in the cells, such as spacers. Long fine tails can often be seen under the microscope behind spacers, evidently caused by a change in particle density in the suspension as it flows around the spacers. These are usually not visible by the naked eye. These observations imply that at least some of the nonuniformities which occur immediately upon filling cells, are a result of flow separation, which causes changes in particle density in the suspension in the affected areas.

A batch of cells was made in which the edges of the glass substrates adjacent to the location of the fill holes were ground to remove the sharp edges. This did not solve the problem, indicating that flow separation is more dependent on movement within the narrow confines of the cell and fill hole, than at the leading edges of the hole.

Electrostatic charges on the walls of the cells could cause particles in the suspension to collect and agglomerate in the vicinity of the charges. The inner surfaces of the glass cells, however, are coated with a film of ITO, which is a fairly good electrical conductor. Experiments were conducted in which both ITO surfaces in test cells were electrically connected together, and connected to the alumimum base plate of the vacuum chamber, which is electrically grounded. The cell was allowed to stand for several hours before filling to dissipate any surface charges. This process appeared to have no effect on the appearance of the cells produced.

Alternate filling methods were examined. Cells were filled by capillary action alone, both in air, using cells with two filling holes on opposite sides of the cells, and in vacuum. Filling by capillary action was recommended by RFI. The results, however, were generally no better than with the vacuum filling method with respect to uniformity, but also had problems with trapped air bubbles. As part of this investigation, empty cells with two fill holes were also sent to RFI for filling in their own facility. RFI was apparently also unable to fill the cells without producing density nonuniformities.

There was much discussion with RFI over the possibility that halocarbon oil was being lost from the fluid in the vacuum chamber despite the presence of the halocarbon oil vapour atmosphere in the chamber. It is this possibility that led to heating of the halocarbon oil in the oil reservoir to raise its vapour pressure, as discussed earlier. The excess vapour pressure, which led to condensation of oil on the chamber walls, eliminated any possiblity that the vapour pressure in the chamber was actually lower than the equilibrium value at room temperature. This

change had no noticeable effect on the quality of the cells produced in the chamber, which indicates that evaporation of halocarbon oil is not likely to be a problem. Nevertheless, all cells were subsequently filled using heated halocarbon oil as part of the regular procedure.

Care was taken to insure that the cell substrates were very clean prior to assembling the cells, these being cleaned in the chemical baths used for glass cleaning in the Active Matrix Liquid Crystal Display development clean room area. However, a possible source of surface contamination was the freon which was, at that time, being used as a medium to spray the spacers onto the glass substrates when assembling the cells. Microscopic examination of clean glass substrates that had been sprayed indicated that there was some contaminating material dissolved in the freon, which was left on the glass in the form of microscopic droplets after the freon evaporated. These droplets did not evaporate away in vacuum. The freon was then replaced with very high purity semiconductor grade acetone for spraying on the spacers, although the lower density of acetone made it harder to keep the spacers in suspension. However, this resulted in no significant reduction in the streakiness of cells. It was also noted that the geometric form of the density nonuniformities in the cells was not generally consistent with the distributrion of surface contamination from the freon in any case. Therefore surface contamination is not believed to be the main cause of the problem.

Another possible source of irregularities in cells is polymer gellation in the suspension. In a series of tests, any clumps of gell which might exist in the suspension were removed by filtering immediately prior to use in filling cells. With the fluids tested, this appeared to improve the quality of cells immediately after filling. However, this did not occur with all fluids (many of which had already been filtered at RFI), and it did not prevent the subsequent appearance of spots in the cells which were originally free of defects, as discussed earlier. Perhaps a process of polymer gelation was continuing inside the filled cells, which was causing particle clustering.

A possible approach to alleviating the effects of agglomeration is to use thicker cells. This would be expected to reduce flow separation, and also help to average out any density variations which did occur. Cells made at RFI are generally several times thicker than those made at LSL, typically 3 or 4 mils (thousandths of an inch) thick. This, of course, results in the operating voltage being several times higher as well, a condition which we have been trying to avoid. Nevertheless, tests were conducted on a series of cells of increasing thickness (about 30, 40, 50, and 60 μm) using suspensions prepared at RFI for these specific thicknesses. Unfortunately, the quality of these suspensions was suspect because even the thinnest cells showed more nonuniformities than such cells normally did, and the thicker ones seemed to be no better. Hence the results were inconclusive. It should be noted, however, that the cleanest looking fluid cells made at LSL were thin 10 μm cells, as reported earlier. This fact appears to be inconsistent with the idea that flow separation will always be greater in thinner cells.

2.2.5 Status of Suspended Particle Fluid Technology

As a result of the work outlined above on SP fluid suspensions, certain conclusions can be drawn about the state-of-the-art of this technology to date, particularly in reference to the VTV application.

SP fluids are cabable of fairly high speed operation, at least as fast as 100 ms. This appears almost instantaneous to the eye, and should be adequate for use in VTVs. Although it was not observed at LSL, RFI claimed to have achieved speeds approaching 20 ms in 3 mil cells using very low viscosity fluid with minimum polymer.

Good contrast has also been demonstrated, even in cells having quite high on-state transmittances. Photopic contrasts greater than 40:1 with on-state transmittances greater than 50% have been measured. Contrasts in the 10:1 range for on-state transmittances of about 70% have also been obtained.

Reasonable operating voltages can also be achieved in thin cells. Operating voltages around 20 volts, for full contrast operation, have been demonstrated in cells of 10 μm cell gap.

Haze is very low when the cells are fully activated. When the cells are dark, measured haze is higher, but is visually less noticeable because of the lower transmittance. Visually, the image seen through a good SP window appears quite clear and haze-free over the full range of optical density.

Therefore, the basic performance parameters of liquid-filled SP cells are good, and should be sufficient to make acceptable VTVs.

However, certain problem areas remain and these must be solved before a practical VTV design can be implemented with this technology. The single most significant problem appears to be inadequate stability of the suspension against clustering or agglomeration of the suspended particles, and inability of any density variations which may occur by flow-separation during filling, or for other reasons, to diffuse away by Brownian motion.

The best performance characteristics of SP cells cannot all be achieved simultaneously, and trade-offs must be made. These trade-offs, however, are generally constrained primarily by the instability problem. To improve stability, the molecular weight of the suspending polymer may be raised, but this generally increases viscosity and reduces operating speed. Attempts to increase operating speed tend to degrade stability and cause more spots and nonuniformities to appear in the cells. The conflict between these two requirements has been observed in the various suspensions received from RFL (On the other hand, it should be noted that with the right polymer this problem could be alleviated because, as the efficiency of the suspending polymer increases, less polymer would be needed. Less polymer could mean increased speed, even though the molecular weight of the more efficient polymer may be higher.) Stability of the suspension also affects the density of particles which it is feasible to maintain in the suspension. Higher particle density is required to achieve a given optical density range in thinner cells, and thinner cells are required to reduce the operating voltage.

Therefore, further development of the suspending polymer system is necessary to increase the isolation of the individual particles from each other, while permitting the polymer-coated particles to move freely around in the suspension under the influence of the Brownian motion. Improvements in this area would greatly enhance the overall performance of the SP fluids.

Another problem with current SP materials is that they are blue in transmission, which is most noticeable when the cells are dark and least noticeable when the cells are light. This is a disadvantage for VTVs, which should be neutral in colour. It is possible that the blue color can be largely neutralized in the future, because not all polyiodide crystals (which are the most efficient particles known for suspended particle applications) are blue. This is an area of development, however, which has not yet been pursued.

A remaining problem with SP materials for the VTV application is that the cells are normally dark when unactivated and become clear only when power is applied. Although this is a basic property of present materials, there is a possibility that this could be reversed in the future. RFI has observed cells become darker as voltage is applied, rather than lighter, in some

experimental materials under certain conditions.

In summary, improvements are required in the SP materials in a number of areas, particularly in the stability of the suspensions and related factors, and in the colour. We believe that the imperfections seen in SP cells to date at LSL are primarily a result of deficiencies in the materials rather than problems with the cell fabrication process per se. To resolve these problems will, therefore, require further development of the chemical system on which the SP material is based.

2.3 Polymer Dispersed Suspended Particle (Film Type) Cells

Near the beginning of 1992 LSL began working with a film version of the suspended particle material, which had been recently invented at RFI.[6] This material represented a breakthrough in SP technology, and promised to solve many of the problems associated with the liquid technology, particularly the instabilities which cause agglomeration. The film material is analogous to Polymer Dispersed Liquid Crystal materials in that tiny capsules of the SP fluid are dispersed in a polymer matrix. It is the separation of suspension into tiny, isolated droplets, each containing a relatively small number of particles, that stabilizes the material against agglomeration. These films are not self-supporting like PDLC films, however, and must be retained in cells similar to those used for SP fluids.

Advantages of SP film cells include the following: films are relatively insensitive to dc voltages, they are not susceptible to agglomeration, even at high fields, there is no settling, enhanced UV stability is easily achieved, hydrostatic pressure in large vertical windows is no problem, and speed can be very fast, probably faster than liquid cells.

Cells made with SP films function in the same way as fluid-filled cells. When the cells are activated, the applied electric field aligns the suspended particles in each capsule, allowing light to be transmitted. When no voltage is applied to the cells, the particles assume random orientations due to Brownian motion, and light is not readily transmitted.

However, the SP films have their own special requirements and problem areas. The refractive index of the material in the capsules (suspending polymer and solvent) must be closely matched to that of the film matrix material in order to minimize the haze produced by scattering of light from the many tiny capsules. It is also more difficult to achieve high contrast in film type cells than in fluid-filled cells because only a fraction of the total volume of the film is active SP material. One objective in making SP films is to maximize this fraction. The capsule size distribution is also important. Film performance is best when the capsule size is small, but not so small that the particles cannot move freely within them. Capsules around 2 μm in diameter are desireable. The formation of capsules depends on an emulsifier, which is also a polymer. The performance of the emulsifier is very important in the production of good films, and development of emulsifiers represented a major area of investigation at RFI throughout the VTV program.

2.3.1 Early Film Material (Swelled Films)

The early films were made by creating an emulsion of crystal concentrate in a type of silicone rubber matrix, and expanding the film with a solvent to provide the suspending medium for the crystals. The crystals were initially received from RFI in the form of a "crystal concentrate" consisting of 2,5-pyrazinedicarboxylic acid polyiodide crystals in isopentyl acetate (IPA). The crystals were then prepared for use in film-making by adding suspending polymer and plasticiser, all in the correct proportions, and removing the IPA. The suspending polymer, which was neopentyl methacrylate/hydroxyethyl methacrylate block copolymer in the form of a

powder was first dissolved in IPA or ethyl acetate to facilitate mixing with the concentrate. This solution was then mixed with the above crystal concentrate along with the plasticizer, which was tri-n-butyl trimellitate. The resulting viscous liquid was sonicated in an ultrasonic bath for about 30 minutes and then poured onto a glass plate and placed in an oven to evaporate the IPA. The resulting material, which was very viscous, similar in appearance to tar, was then scraped from the glass and stored in a small glass bottle. It was this material which was the final "crystal concentrate" used to make the actual films.

The matrix material in these films was dihydroxy-terminated polydimethylsiloxane, which is the simplest basic silicone. This polymer has the form of a viscous liquid. The emulsifier used to create the capsules of SP fluid was polydimethylsiloxane/neopentyl methacrylate block copolymer. Actually a succession of different emulsifiers was used, but this one represented an improvement over others at the time, and was subsequently used extensively. A crosslinker was used to crosslink the matrix polymer to form the final film. The crosslinker was tetra-n-butylorthosilicate. A catalyst, dibutyltindilaurate, was also used to catalyse the crosslinking reaction.

The film material was made by weighing and mixing together, one at a time, the matrix polymer, emulsifier, crystal concentrate, catalyst and crosslinker. Mixing was done in a small beaker using a high speed homogenizer. The resulting material, which was an emulsion of SP suspension in matrix polymer, was then vacuum degassed. Finally, it was poured or layed down with a spatula along one edge of a sheet of flexible plastic, such as mylar, and spread out into a film using a doctor blade or equivalent tool. The film thickness could be varied by adjusting the spacing under the blade. Films were usually made a few thousandths of an inch thick. The plastic sheet with the SP film on it was then baked in an oven to accelerate the crosslinking reaction and cure the film. When cured, the film had the consistency of a very flexible but rather weak silicone rubber.

By the end of May 1992, a significant change to the film chemistry was made by the introduction of a matrix polymer/emulsifier in a single chemical. RFI referred to this material as a crosslinkable emulsifier. It consisted of a silicon atom with polydimethylsiloxane polymers on each of three bonds, and polyneopentyl methacrylate on the fourth bond. Films made with this material initially included matrix polymer, but were later made without using any matrix polymer. Otherwise, the film-making process was the same as for previous nonswelled films. Much work was done at LSL using this material.

2.3.1.1 Cell Fabrication

SP cells for PDSP films are essentially the same as for fluid cells, employing two ITO-coated transparent substrates and a means of maintaining the desired cell gap. However, they are assembled in a different manner.

To complete a cell using expanded SP film, a piece of film the appropriate size was cut out by cutting the plastic sheet carrying the film. Allowance had to be made for a linear expansion to 170% of original size which would occur when a swelling agent (or solvent) was subsequently applied. Although many different solvents were tried, the one normally used was 3,6-dimethyloctane. The dimethyloctane was spread liberally over one of the ITO-coated glass substrates used to make the cell, and the film, still on its plastic backing, was then placed film side down on this wet surface. As the film absorbed the dimethyloctane, it would swell and buckle and gradually separate completely from the plastic backing. With practice, the film could be made to spread out over the substrate without wrinkles and with minimum entrapped air bubbles as it swelled and separated from the plastic sheet. The plastic sheet was then removed, more dimethyloctane was spread on top of the film, and the second glass substrate was placed

on top to complete the cell. Excess solvent, and usually any air bubbles caught between the film and substrates, could be squeezed out by pressing the substrates together. The film itself acted as the spacer for the cells, so that additional spacers were not required. Finally, the cell was sealed around the edges with epoxy to hold the cell together and prevent the solvent from evaporating away.

As the solvent swelled the film it also entered the capsules containing SP crystals and polymer, and provided the solvent necessary to complete the suspension. Prior to swelling with solvent, this type of film showed no Brownian motion, and would not operate as a light shutter. After swelling, the particles in the capsules exhibited Brownian motion, and the film became electrooptically functional. In fact, the Brownian motion in the capsules of some of these films was the most spectacularly evident of that seen in any previous SP materials examined.

2.3.1.2 Film Performance and Problem Areas

An advantage of these films appeared to be, as mentioned previously, that they are more stable than the liquid suspension against degradation due to agglomeration. Once made, the films did not change significantly with time, provided there was no loss of solvent by evaporation. (As discussed later, however, this did not remain the case with all films.)

Switching speeds of these early films were very fast. The highest speeds were reported by RFI, with some films reaching close to 20 ms. This was equal to the very fastest fluid cells, as reported by RFI, and was significantly faster than most fluid cells.

Contrast, on the other hand, was much poorer than for the best liquid suspensions, being typically around 5:1.

These early films certainly worked, but their optical quality was very poor. Optical density was usually very non-uniform, with both large scale and small scale variations occuring over the entire cell area. The large scale variations were primarily caused by variations in film thickness produced during the film spreading process. The small scale variations consisted of dark spots, streaks, and often a grainy mottled-appearing texture. Many experiments were conducted to develop and perfect the fabrication process in order to improve the quality. Process parameters, including mixing proportions, were varied widely during both film and cell making. Changes in film composition were also made from time to time as a result of continuing developments at RFI.

It was determined that many factors were involved in producing these problems, aside from the nonoptimized chemistry of the films themselves. Because the films were thin, being only a few mils in thickness, it was essential to use high precision tools to spread the film in order to avoid significant film thickness variations. It was found that the edge of the doctor blade, or spreading bar, had to be highly polished in order to prevent the appearance of fine steaks due to irregularities or scratches in the edge of the blade. The blade also had to be very straight, and the substrate on which the film was formed had to be very flat. Ultimately, a 1 inch thick plate of pyrex glass, polished to 2λ flatness was used as the substrate on which to spread the films, and a 1/2 x 1 inch tool steel bar ground straight, with a lapped edge, was used as the spreading bar. The required gap between the bar and the plate was maintained by taping two small pieces of shim material around the lapped edge of the bar, a suitable distance apart, to act as spacers between the bar and the glass. The bar was held tipped at about 30° - 45° to the glass surface when spreading the film.

Film thickness depended on both the drawing speed of the bar and the tilt angle, as well as the viscosity of the material. Because the films were spread by hand, there were always some

variations in density due to nonuniform motion of the bar, or variations in the tilt angle. With practice, these could be minimized, and were not an immediate concern because their origin was well understood and could be eliminated with the proper equipment.

Rolling the film material onto the working substrate was also tried, but this was unsuccessful because the tackiness of the material resulted in severe textured patterns in the surface of the rolled film.

Spreading films directly onto glass plates, rather than onto plastic sheets was investigated, to avoid the added problems of the plastic. However, whereas the films would separate from ordinary plate glass when the dimethyloctane solvent was applied, they usually would not separate from the surface of the polished pyrex. Evidently chemical bonding was taking place, possibly between silanol groups on the glass surface and OH groups of the siloxane chain, or the crosslinking agent. The plate glass, however, was not flat enough to give sufficiently uniform film thickness. Handling the swelled film and transferring it from one glass plate to another (the cell substrate) without damaging or tearing it was also difficult and impractical.

Therefore, plastic sheets were again used as substrates on which to form the films. Many different types of plastic sheets, of different thicknesses were tried as substrates. Many were ruled out because they could not be made to lie flat on the pyrex base plate, or their thickness uniformity, surface flatness and/or smoothness was not good enough. Some plastics, notably Lexan, reacted with the SP film material. Best results were achieved with polished 3 mil mylar sheets. In order to achieve good flatness, the mylar was "glued" to the pyrex plate by placing a few drops of deionized water on the glass, and then rolling the mylar onto the surface with a semi-hard rubber roller (photographic roller). Air bubbles and all excess water were rolled out until the plastic made virtual optical contact with the glass and minimum interference fringes were seen. The film was then spread on the plastic sheet immediately, before it began to separate from the glass again. The entire pyrex plate, with the mylar sheet and the SP film were then placed in an oven to cure the film.

The success of this method also depended critically on cleanliness of the glass and plastic surfaces. Particles as small as a few microns in diameter between the plastic and the glass would cause bumps in the plastic and cause visible defects in the films. These defects had the form of light spots, caused by the decreased thickness of the film. It was therefore necessary to perform all of these experiments inside a clean room to avoid stray particles.

The problem of dark spots in the films was investigated at length. It was determined that these spots were caused by small contaminating particles, such as tiny fibers, in the film material itself. After the films were spread, the film material appeared to wick up around these particles, forming small mounds of material higher than the surrounding film. The increased thickness made the film appear darker in these areas, thereby producing dark spots. Sometimes the dark spots were surrounded by a light concentric ring.

Even though the operation was moved into a clean room, and care was taken to insure that there were no particles on the surface of the mylar before spreading the film, the dark spots persisted. It became evident that the chemicals themselves were contaminated with foreign particles. Filtering the polydimethylsiloxane in a vacuum filter apparatus, confirmed that it contained many stray particles, mainly small fibers. Thereafter, this material was vacuum filtered before making any films, and the incidence of dark spots decreased significantly, though not totally.

One of the remaining problems evident with the film quality was the appearance of an irregular mottled or grainy structure similar to the coarse grain sometimes seen in photographic

films. The graininess was caused by deficiencies in the crosslinkable emulsifier. This is one of the first crosslinkable emulsifiers produced, and was synthesized using the rather unsophisticated free radical method of polymer synthesis. The result was an impure mix of molecular variants, and so it was far from optimum. Some of the molecules did not have the correct terminations, and dissolved in the capsules at both ends. This tended to pull the capsules together, causing clumping, and hence the appearance of graininess.

It was anticipated that improvements in the crosslinkable emulsifier could be achieved by using the group transfer method of synthesis, which is much more specific with respect to the molecular species produced. However, it was not feasible at the time to implement this procedure due to limitations in available facilities and equipment.

Another problem with film quality was a high level of haze. This was a result of mismatches in the refractive indices of the suspending polymer, matrix polymer, and solvent. The refractive index of the suspending polymer was much higher than that of the other materials. Achieving a close match in the indices of the materials, while simultaneously optimizing their functional performance was a continual challenge for RFI throughout the entire period of film development work.

Although the overall appearance of completed cells (with respect to visual defects) did not change with time, most of the cells gradually lost contrast until they ceased working altogether. This is believed to be a result of the loss of the solvent from the cells. This occurred despite the cells being sealed with epoxy. Evidently the solvent either difuses through the epoxy, or escapes through tiny leaks.

Film quality improved in many respects as a result of this work, notably in the reduction of discrete process-related defects. However, film quality and performance remained inadequate to make acceptable VTVs as a result of residual process problems which could only be eliminated in a well designed production process, and problems with the chemistry of the films, which required more research to resolve.

2.3.1.3 VTV Experiments with Swelled Films

Once the ability had been achieved at LSL to make sufficiently large films, experiments were conducted to investigate the feasibility of installing such a film on the curved surface of a polycarbonate visor. Considering the mechanical difficulties involved in making high quality uniform films on a flat surface, it was believed that to form such a film on a curved surface, equivalent to a visor, would be very difficult, and would require specially designed precision equipment. Such a process would not be feasible in the context of this program.

Hence films were made on flat mylar sheets, swelled, and transferred to the curved surface. Preliminary experiments were conducted using convex lenses to provide the curved surfaces. These tests were essentially successful, except for minor problems. Tests were also conducted to verify the compatibility of the SP film material with polycarbonate, and no problems were discovered. Tests then proceeded with a real visor. To do this the visor was placed, convex side up in a glass tray, and dimethyloctane was poured over the surface. The mylar sheet with the SP film was then soaked in dimethyloctane as well, turned over and placed on top of the visor. The surfaces of the film and the visor were then kept soaked with the solvent with the aid of a large eye dropper, until the film completely separated from the mylar sheet and settled down onto the visor. Care had to be taken to prevent the film from sliding off the visor, which was very slippery when there was an excess of solvent between the two surfaces.

When the film was in place on the visor, it had large wrinkles around the edges. It was found, however, that the film would stretch just enough without tearing to remove the wrinkles and allow it to conform to the curvature of the visor surface. If it was released, it would then pull back and the wrinkles would reform. Stretching the film to conform to a curved surface must, of course, result in variations in film thickness over the area. Those areas of the film which are stretched the most become the thinnest. However, these variations were not evident visually in the test films, at least when unactivated, and so may not represent a significant problem. It is also possible, however, that such variations could have been obscurred by the overall poor optical quality of the film.

It was not possible, with the available visors, to place one visor over the other and press them together to make a cell in which the dimethyloctane would wet the second visor over its entire surface. The reason for this is that the curvatures were too different, and the visors were too stiff to force them to conform to each other.

The film was held in place on the convex visor surface and allowed to dry, after which it remained attached to the visor. As the solvent evaporated, however, the film started to shrink again back to its dry size, and this caused it to split near the edges of the visor.

Although an assembled visor could not be made with the components available at the time, these experiments demonstrated that it is possible to apply a flat SP film to a curved visor surface without tearing it, so that it should be possible to make a working VTV using this type of SP film material.

2.3.2 Non-Swelled Film

A very significant breakthrough in SP film technology was made at RFI near the end of 1992, which resulted in what might be called the Second Generation Film Material.[6] LSL began working with this material in February 1993, and all SP films made at LSL from that time to the present were of the new type. The advance consisted of eliminating the need for swelling the film with a solvent, and was based primarily on a new crosslinkable emulsifier.

Advantages of the new type of SP films include the following. The film-making process was greatly simplified by eliminating the need to make the films separately from the assembly of the cells, by eliminating the messy and difficult process of swelling the films with an organic solvent and then transferring them to the cell substrates, and by eliminating the need for liquid (solvent) inside the cells and the need to prevent it from evaporating with time.

It was no longer necessary to form the films by spreading them onto a flat substrate using a doctor blade, or spreading bar. The films could be formed directly in the final cells, as described below. A very significant advantage which resulted from this change is that both surfaces of the film could be formed directly by contact with a smooth substrate, rather than only one. This eliminated the surface irregularities, which always arise from the film spreading process, as a source of optical defects in the films.

The non-swelled films will also accept a higher concentration of crystals than the earlier swelled films. These films can therefore be made darker for the same thickness, and can achieve a higher contrast ratio.

Haze has also been greatly reduced in the new films compared to the swelled films. For the first time, film type cells can be made which look very clear visually.

2.3.2.1 Preparation of Films

To make the new SP films, the matrix polymer, crosslinkable emulsifier, suspending polymer, crystal suspension, catalyst, and crosslinker were mixed and vacuum degassed in a similar manner to the earlier swelled films. In this case, however, all the chemicals, except the crosslinker and catalyst, could be mixed together at the same time, rather than all in individual sequence as previously. These were mixed in separately after the other components were mixed. Once the film material was completely mixed, spacers were added and mixed in by hand. The spacers used for most of the cells made with this material were glass spheres of 31.5 μm diameter. When films of this material were made and cured by baking they immediately became functional as light shutters without further processing.

2.3.2.2 Cell Fabrication

SP cells using non-swelled films are, again, essentially the same as for fluid cells, employing two ITO-coated transparent substrates, as well as spacers to establish the desired cell gap. However, again, they are assembled in a different manner. The film material is generally much more viscous than the fluids, and film cells are assembled with the film material in place. The film material, immediately after mixing and degassing, is spread over one of the substrates in a layer thicker than the final film thickness. As stated previously, the spacers required to establish the cell gap are premixed into the film material. The second substrate of the cell is then pressed onto the first substrate and the excess material is squeezed out until the final cell gap is achieved. The assembled cell is then placed in an oven to cure the film inside the cell. The cell may then be cleaned around the edges and sealed with epoxy. Unlike fluid-filled cells, or even swelled film cells, the expoxy is not necessary to hold the cell together, but only to seal and stabilize it. The film itself, being a rubber-like solid at this point, holds the cell together.

This process works reasonably well only for small cells about 2 inches square or less. The reason is that air bubbles become trapped in the film material as the upper substrate is lowered onto it, even when contact with the film material is made initially along one edge and the substrate is gradually rotated down into position to create a single advancing line of contact between film and substrate. The larger the cells, the more difficult it is to avoid air entrapment. In the case of visors, the difficulties of cell assembly are compounded by the curved shape of the mating substrate surfaces.

To avoid this difficulty a method was developed to permit cell assembly to be accomplished in vacuum. In this method, the film material is spread in a layer over one substrate (the "lower" substrate) as in the previous case, except that for larger cells the layer thickness and uniformity must be much more accurately controlled. This is accomplished using a doctor blade to spread the material. A small bead of extra material is also placed around the edges of the coated area using a syringe. It is not necessary to degas the film material first. The coated substrate is then placed coated side up on a fixture inside the vacuum chamber. The second substrate (the "upper" substrate) is then suspended, ITO side down, about 1 cm above the lower substrate by means of an electromagnet acting on a magnetic plate fastened temporarily to the back of the substrate with adhesive tape. The position of the upper substrate is carefully aligned visually with respect to the lower substrate, taking into account the parallax between the two plates. The chamber is then closed, and pumped down to less than 1 mT pressure. In this procedure, the film material, which is spread out in a thin layer over the lower substrate, becomes degassed during the pumping cycle.

After pumping is complete, the electromagnet is deenergized, releasing the upper substrate. This substrate falls straight down to the lower substrate and contacts the raised lip of material spread around the edges of the latter substrate. This forms a seal around the cell, that

prevents air from entering the cell when the chamber is vented to the atmosphere. If irregularities in the bead height prevent a complete continuous seal from forming, a mechanical manipulator inside the vacuum chamber is used to press down on the upper substrate. In this way any gaps in the seal can be closed. It is evident when the seal is complete because the areas where the film material has wet the glass substrate are clearly visible.

Air is then vented into the vacuum chamber, and the pressure forces the two substrates together until there are no remaining voids inside the cell. The cell is then removed from the chamber. At this point the cell is optically very nonuniform due to variations in cell thickness. In particular, the edges are generally much darker than the central area because of the extra material placed there.

In order to bring the entire cell down to the same uniform thickness, determined by the spacers in the film material, it must be subjected to uniform pressure over the entire cell area and the excess film material allowed to flow out. This is accomplished by placing the cell inside a vacuum bag. A method which has proven to be successful is to use a meat packing machine, which is specifically designed to seal material inside vacuum bags. Atmospheric pressure then provides highly uniform pressure over the cell area. However, the cell will not "clear" (achieve final controlled cell gap) unless the edge effects caused by the curvature of the bag around the edges of the cell are eliminated, and clearance space is provided to permit the excess film material to flow freely out of the cell. These two problems are solved by placing the cell inside a plastic frame having the same thickness as the cell, and having a channel or groove running around the inner edge and "radial" channels running from this groove to the outer edges of the frame. The radial channels do not break the surfaces of the frame. The frame supports the vacuum bag around the edges of the cell so there is no wrap-around effect, and the channels provide space into which the excess material can flow.

Cells as large as 4 x 7 inches were made by this process. Clearing of 4 x 7 inch cells requires typically 10 to 30 minutes depending on the viscosity of the material and the size of the cell. As the process proceeds, the edges become light and the centre darker, the reverse of the original pattern. Then the centre gradually clears until the entire cell is uniform. The cell, still in the vacuum bag, is then placed in an oven to cure the film material.

Clearly, the viscosity of the film material is fairly critical in this process. After the film material is mixed, it immediately begins to cure, even at room temperature. As it cures, the viscosity increases. If the viscosity of the material is too high initially, then there will be insufficient time to assemble the cell, pack it in the vacuum bag, and allow it to clear before the material becomes too thick to flow. This is a problem which we encountered often with the available film materials.

High viscosity of the film material is expected to be only a temporary problem with this material. Better control and stabilization of the molecular weight of the emulsifier should result in much lower viscosity pre-cure film material. If the viscosity can be reduced sufficiently, then it may well be possible to vacuum fill preassembled cells of visor size with film material in the same way that fluid cells are currently filled. This would be a considerable advantage over the present method. Vacuum filling of a small (2" x 2") cell with a faily low viscosity film material has already been successfully demonstrated at LSL.

2.3.2.3 Film Performance

The inherent advantages of the non-swelled films, discussed above, led to certain improvements in cell quality over the earlier films. In particular, the fact that both surfaces of the film were formed by the smooth surfaces of the two cell substrates, prevented the bumps

that occurred in spread films due to foreign particles in the film material. Even though foreign particles in the chemicals still had not been eliminated, they simply became imbedded in the final film, resulting in minor visual specks. Hence, the problem of dark spots due to dust particles in the film material were largely eliminated.

Streaks due to irregularities in the earlier film spreading process were also virtually eliminated, provided due care was taken in controlling the average thickness of the film spread on the "lower" substrate before assembling the cell. If irregularities were too great, or the film was too thick, then residual marks would be left in the cell after pressing the cell in the vacuum bag. By using good technique, chemically cleaned substrates, and vacuum assembly, 4" x 7" cells were fabricated, using some batches of film material, which were extremely uniform with virtually no visible spots, streaks, or other density non-uniformities.

These were unquestionably the best looking SP cells that had been made at LSL, either liquid or film type. A great deal of effort, and many experiments were required to achieve these results, but they clearly demonstrated that SP film cells of excellent optical quality could be made in sizes large enough to fabricate VTVs.

Great improvements have also been made in refractive index matching by adding phenyl groups to the suspending polymer. This has greatly reduced the haze seen in earlier films.

Threshold characteristics of the unswelled film cells are similar to those of fluid-filled cells. Figure 2-6 shows a typical threshold characteristic curve for a film cell (31.5 μm thick) at 0° inclination angle. It is seen that the characteristic is fairly linear over quite a large range of both voltage and transmittance, giving good grey scale control.[7]
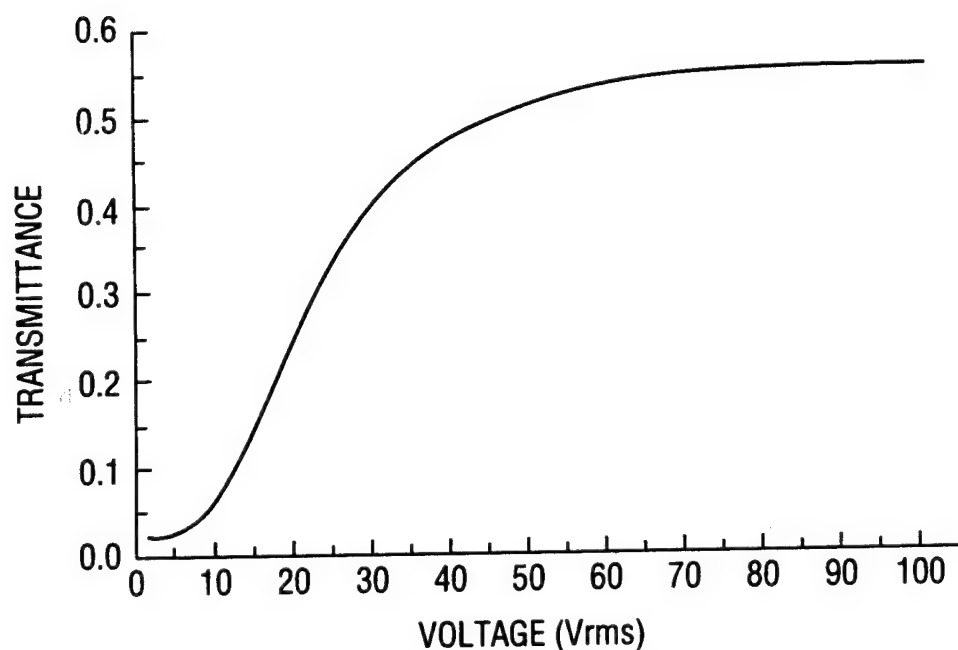

Figure 2-6. Threshold characteristic of a PDSP cell for 0° inclination angle.

Figure 2-7 shows isoinclination curves of transmittance for 15º, 30º, 45º, and 60º inclination angles (angles measured from the normal to the test cell surface), for zero drive voltage. On-axis (zero inclination angle) transmittance, which is not shown, is 2.2 percent. Transmittance decreases with increasing inclination angle, but is virtually independent of azimuth. The small variation with azimuth which does occur is not believed to be a fundamental property of the SP shutter, and is probably a result of nonuniformities in cell thickness and imperfect mixing of the film material.
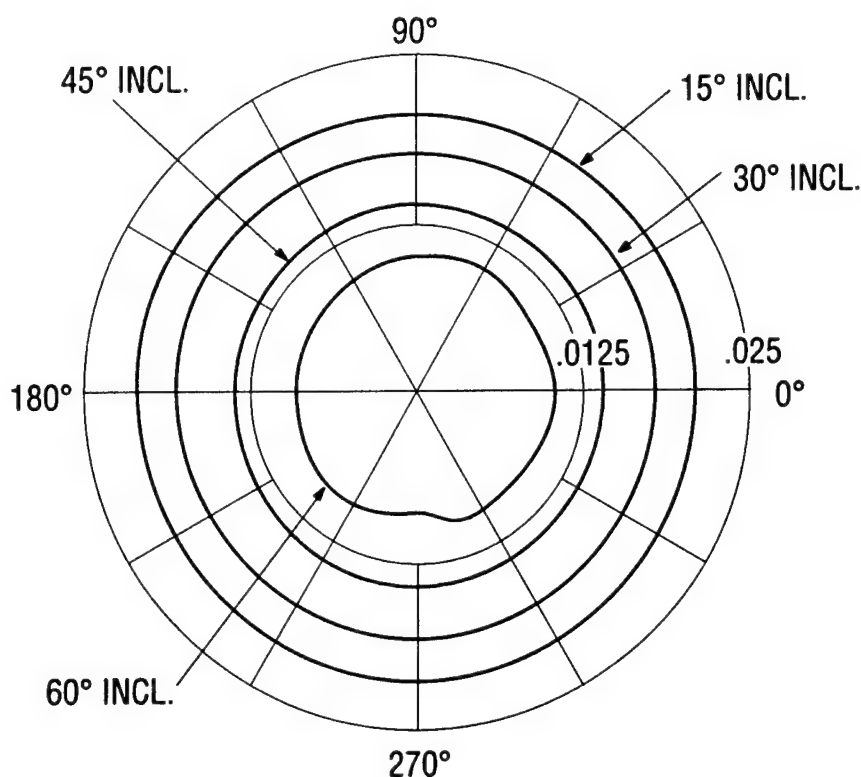

Figure 2-7. Isoinclination curves of transmittance of a PDSP cell in the quiescent state.

Similar isoinclination curves are shown in Figure 2-8 for maximum transmittance, obtained at 100 volts excitation. The transmittance at 0º inclination angle is 55 percent. Again, the transmittance decreases with increasing inclination angle, and is substantially independent of azimuth. The minor variations in transmittance with azimuth angle seen in these curves are consistent with nonuniformities in the cell as the possible cause.

Figure 2-9 shows isoinclination plots of the contrast ratio of the test cell. Contrast ratio decreases from approximately 25:1 at 0º inclination angle to 21:1 at 15º inclination and approximately 17:1 at 60º inclination. Again, contrast is essentially independent of azimuth. It should be noted that this is an advantage of SP cells over TN liquid crystal cells, for example, in which the contrast does vary with azimuth. Furthermore, the decrease in contrast ratio with increasing inclination angle is much smaller than it is for TN cells. Therefore, SP cells have much

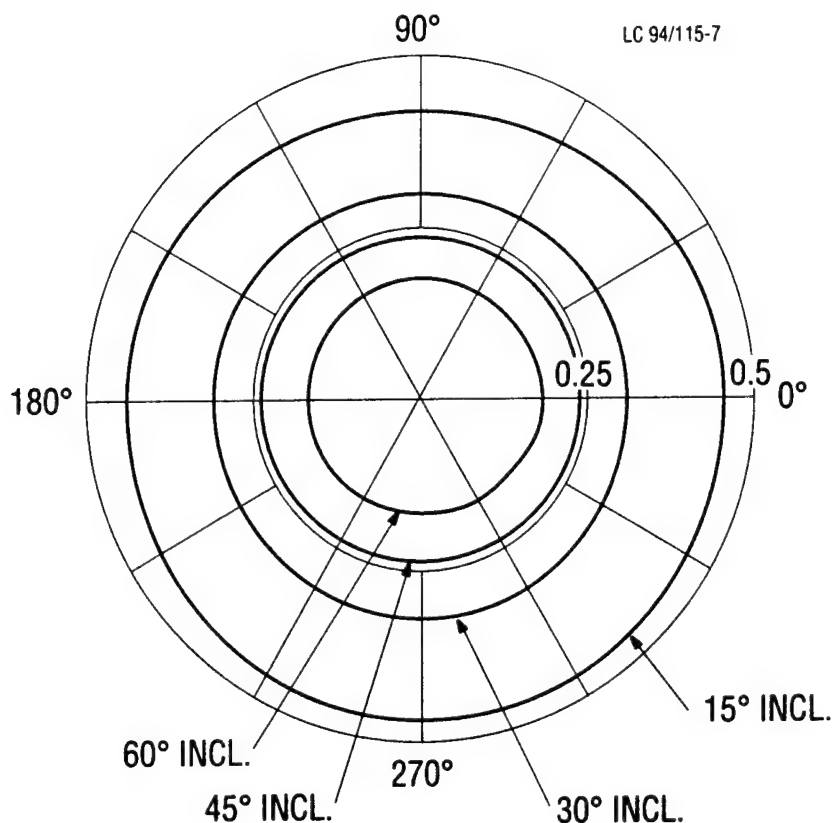wider viewing angles, as well as higher transmittances than TN cells.

Figure 2-8. Isoinclination curves of transmittance of a PDSP cell when fully turned on.

Figure 2-10 shows the variation of haze as a function of excitation voltage. Haze is highest with the cell in the off state, and decreases with increasing voltage. In the on state, the haze becomes essentially zero on the curve.

Spectral transmittances in the off and on states are given in Figure 2-11. Note the different vertical scales on the two curves, which exaggerates the variation in off response compared to on response. Nevertheless, these spectra show a large peak in blue transmittance at about 410 nm in the nonactivated cell. Contrast is minimum in the blue, and maximum in the red, at about 650 nm.

2.3.2.4 Problem Areas

However, there were also a number of problems with the new film material which, to date, have prevented the fabrication of a good quality VTV. One of the most serious problems is the formation of "crazing lines" in completed cells. These are visible marks in the form of fine branched lines resembling ferns, which usually grow in from near the edges of the cells. They often begin as short, irregular lines, or clusters of such lines near the corners of the cells, or along the edges. Over a period of days or weeks they become progressively more extensive,

usually fanning inward. In some severe cases they have occured in the central area of cells as well. As seen in reflected light they have the visual appearance of areas where separation of the film from the ITO-coated glass substrate has occured. However, for any particular crazing line, this "separation" occurs from only one substate at a time. In other words, the pattern of crazing lines is different as seen on opposite sides of the cell.
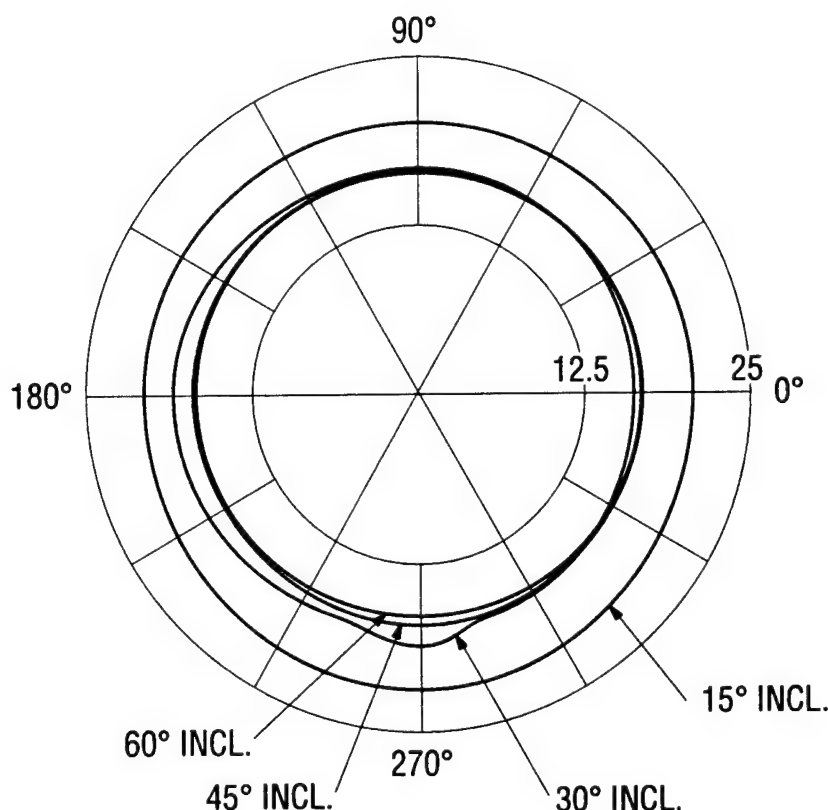

Figure 2-9. Isoinclination curves of contrast ratio.

In transmitted light, however, they all appear dark against the background of the cell, and they do not become significantly lighter when the cell is energized. They remain dark even when the cell voltage is raised to much higher than normal values, i.e. to values much higher than that required to fully turn on the rest of the cell. This demonstrates that the dark appearance of the lines is not simply a result of a layer of air or gas of low dielectric constant lying between the film and the substrate, thereby reducing the electric field in the film itself. Hence, the crazing lines must represent areas where the film itself no longer functions properly.

To date, the cause of these defects has not been determined, although it may be related to the fact that the film shrinks 10-15% on curing and is therefore subjected to tensile stress inside the cell where it is held in place by the cell walls. It has been established that the occurence of crazing lines can be signficantly reduced by cutting the vacuum bag holding the cell prior to placing it in the oven to cure the film. This releases the pressure and allows the cell to relax. If this is not done, cells which look perfect prior to baking, and even subsequent to baking before the vacuum bag is cut to release the cured cell, usually develop crazing lines

shortly after being released. The highly uniform 4" x 7" cells mentioned above were made by releasing the pressure on the cells before baking. Even so, it has been found that some test cells which were free of crazing lines for many months after being made, eventually started to show these defects around the edges. This problem remains to be solved, and it is still not clear to what extent it is a process problem and to what extent it is a chemistry problem.
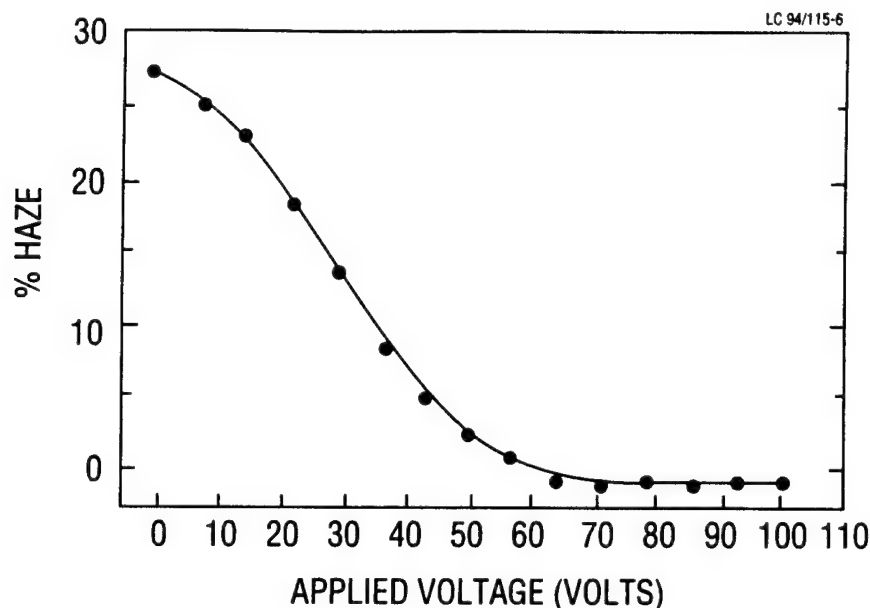

Figure 2-10. Percent haze of a PDSP cell.

Another significant difficulty with the new unswelled films is that the electrooptical performance degrades with time. Many test cells at LSL have shown the same behaviour, including the good 4" x 7" cells mentioned previously. Actually, there appear to be two effects which may or may not be related. In one case, many of the cells developed a zone or "border" around the edges that remained darker than the rest of the cell when energized. Usually these cells would come back to a uniform density when turned off, but when turned on, the border area would remain dark. This effect occured sometimes on the first excitation of the cell after completion, and sometimes after several subsequent excitations. Once formed, it remained permanent, and gradually increased in width. In some cells a series of concentric "rings" appeared around the border area on subsequent excitations about a day apart. It did not matter whether the cells were sealed around the edges with epoxy or not.

The cause of this problem was eventually identified to be at least partly a loss of crosslinker in the vacuum during cell assembly. Small test cells assembled in air, with only minimal vacuum degassing of the material, showed a marked reduction in the formation of dark rings after excitation. However, large cells of good uniformity could not be assembled without the vacuum assembly process, because of the problem of entrapped air. To properly solve the problem an alternative crosslinker with very low vapour pressure, which would tolerate exposure to vacuum without evaporation, was required. Such a chemical was identified (tetra 2-ethylhexy-

lorthosilicate) and ordered from a chemical supply house. Although this has now been received, it has not yet been tested due to the current unavailability of the required film chemicals from RFI.
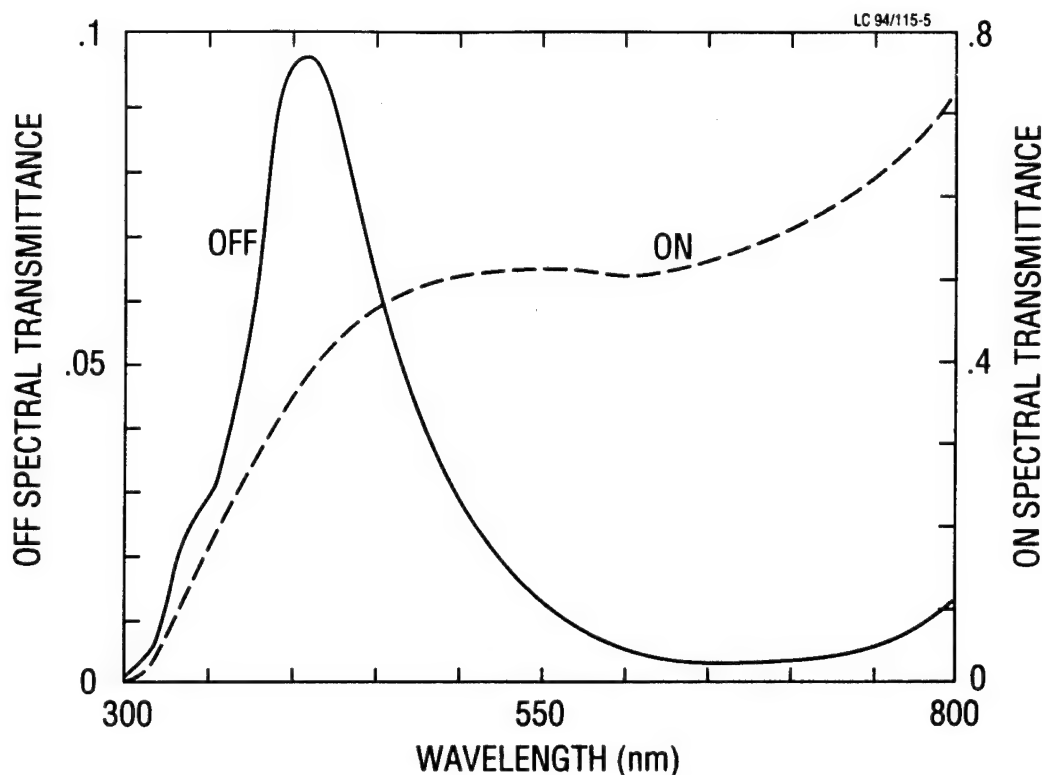

Figure 2-11. On- and Off-state spectral transmittances.

The second effect is a longer term degradation of contrast ratio with time, which generally occurs predominantly in the central regions of the cells. Improvements in the suspending polymer at RFI late in the summer of 1993 alleviated this problem to a significant extent, though not completely. It is thought that a crosslinking reaction was taking place in the suspending polymer. Because it is the central areas of the cells which deteriorate more rapidly than the edges, it is conjectured that some material that causes the film to slowly degrade is escaping the film around the edges more rapidly than in the centre. The only volatile chemicals used in the film are the catalyst and crosslinker. However, sealing the cells with epoxy does not appear to eliminate the problem. Further work is needed in this area to extend the life of the cells.

However, it is important to note that RFI has a few cells which are now more than one year old, which still exhibit virtually the same contrast ratio as they did when they were made. Unfortunately, these cells are very slow, which may partly explain why they have not deteriorated. Nevertheless, they indicate that stable cells can be produced, and a viable solution to the degradation problem can be expected in the future.

The non-swelled films are, to date, significantly slower than the swelled ones. This, however, is recognized as a problem of excessive polymer viscosity. Further improvement of the polymers is expected to result in faster films.

Operating voltages are rather high on the nonswelled film cells, which is a result of making the cells thick to achieve high contrast. Voltages to fully turn on 31.5 μm cells are typically close to 100 V.

2.3.2.5 Prototype VTV Experiments Using Goggles

In order to fabricate a SP VTV two mating visor parts are required to make the SP cell, for either fluid suspension or film. Mating pairs of visors, however, are not available and would have to be custom made. This would involve optical design, and the making of molds. The cost of making mating visors is, therefore, beyond the range of the present program.

Discussions of this problem with Gentex Corporation, Pennsylvania, which is a supplier of visors for the US Air Force, led to the generous offer of samples of mating pairs of goggles which could be used for prototype feasibility tests. These goggles are made of polycarbonate plastic, as are the visors, and have a similar radius of curvature. However, they are smaller, being designed as sun/wind/dust goggles, and were used extensively in the Gulf War. Only three pairs of colorless, mating parts were available as rejects from an earlier production run, with only minor cosmetic defects. These parts were supplied to LSL at no charge.

2.3.2.5.1 Goggle Coating

Coating polycarbonate with ITO is a difficult problem because of the high temperatures involved in the coating process. Experiments in coating polycarbonate (Lexan) sheets with ITO at LSL gave unsatisfactory results. The problem was discussed with outside suppliers, and coatings were finally ordered from Southwall Technologies, Palo Alto, California. These coatings were not actually ITO, however, but consisted of dielectric-metal-dielectric layers. Transmittance achievable was about 80%. Tests on a sample piece of coated polycarbonate showed that there was no detectable reaction between the coating and the SP materials.

2.3.2.5.2 Goggle Assembly

The vacuum cell assembly process described earlier for unswelled films was further developed to permit goggle cells to be assembled, and a special curved frame to use around the goggles in the vacuum bag to reduce edge stresses and allow the escape of excess SP material was made. Several attempts were then made to fabricate SP goggles using the unswelled film material, and using the same procedures used to make the uniform 4" x 7" glass cells.

2.3.2.5.3 Goggle Quality and Problem Areas

Several of these goggle cells were assembled successfully and pressed to uniform thickness in vacuum bags, resulting in cells which initially looked smooth and uniform. It appeared, though, that uniformity was achieved with little margin for error with respect to time, because the viscosity of the available film polymers had increased enough to significantly slow the flow of excess material out of the cells. After the goggles were baked to cure the film, however, severe degradation occurred in every case, mainly in the form of crazing lines. Cutting the vacuum bag to permit the cell to relax before curing, did not prevent the appearance of the crazing. It is possible that stresses put on the film by movement of the cell walls under the heating and cooling cycle damage the film and cause the crazing.

Another potential area of difficulty is inadequate cleanliness of the goggle surfaces. The goggles will not tolerate the chemical cleaning process regularly used at LSL to clean glass substrates for liquid crystal displays. This was verified by tests performed on small samples of polycarbonate sheet. Therefore, cleaning was done using a strong detergent solution in deionized water, and a solvent, such as isopropyl alcohol, which would not not attack the polycarbonate. In the first attempt to make goggle cells the goggles were used directly as received from the coater without touching the surfaces except to blow off any loose dust with dry nitrogen. Subsequent trys, however, required removal of the film material from the surfaces, followed by cleaning as above. It is suspected, though not proven, that surface contamination left by the cleaning process might have contributed to the poor optical quality of the assembled goggle cells.

These experiments have clearly demonstrated that unswelled SP film cells made with curved goggles, similar to visors except for size, can be made with smooth and uniform appearance, at least up to the film curing stage. The problems which arose during or after curing are fundamentally the same problems seen in flat glass cells, and are not unique to the goggles. The fact that the goggle cells were poorer looking than the flat glass cells was probably a result, at least partly, of the occurance of greater stresses on the film in the goggles than in flat glass cells and poorer surface preparation.

Future improvements in the film chemistry, and possibly in processing methods, should alleviate the present difficulties in making high quality cells with good stability and longevity. It should then be possible to make working SP VTVs using this technology. It is difficult at this time, however, to predict how long it will take to achieve this goal.

2.3.2.6 Self-Supporting Suspended Particle Films

Some preliminary experiments were conducted in an attempt to make encapsulated suspended particle films strong enough to be self-supporting, rather like plastic films. Many combinations and proportions of mixtures were tried using epoxy and polyvinyl alcohol (PVA) as matrix materials. Some success was achieved in PVA, in which the suspended particle concentrate was found to be immiscible and formed droplets under certain conditions. A transmittance range of 5% to 18%, for a contrast ratio of 3.6:1 was measured on one test cell. The film could be peeled from the glass substrate on which it was made, and had the consistency of a plastic film similar to cellophane. This film would still work when sandwiched between ITO-coated glass plates.

The optical quality of these films was extremely poor, with very non-uniform density and high haze levels. It is recognized that there are serious technical problems which would have to be solved to make useable, good quality films of this type. A uniform dispersion of SP droplets in a tough plastic film must be produced, while simultaneously achieving a high degree of refractive index matching between the film and the droplets. Present materials have substantially different refractive indices. Nevertheless, we believe that this work shows that it is possible to make plastic films of encapsulated suspended particle material.

2.4 Conclusions

Three classes of suspended particle materials have been investigated at Litton Systems as possible technologies for use in Variable Transmittance Visors: fluid suspensions, swelled films, and nonswelled films. Processes have been developed to make light shutters using each of these materials, and many experiments have been conducted on each of them to evaluate and improve on their performance.

Fluid cells function well, giving good contrast and speed, well within the ranges required for a usable VTV. However, it has not been possible, to date, to make cells free of defects in optical density in the form of spots and streaks. Although it is possible that this problem might still be partly process related, we believe that it is primarily a result of material deficiencies. Further improvements are required in the suspension system to increase the stability of the suspension against clustering and agglomeration of the particles, and to permit any density variations that may occur as a result of phenomena such as flow separation during cell filling to spontaneously diffuse away by virtue of the Brownian motion of the particles. We do not doubt that this result can be achieved with further development of the material. When this is accomplished, a high quality VTV could certainly be made.

Cells made using swelled films also showed good speed, but less contrast than fluid cells. Optical quality of these films was distinctly worse than the best of the fluid cells, however, and was largely process related. These films are formed by spreading the material on a substrate which does not become part of the cell itself. It was found that to achieve films having very uniform density, very precise control of the film-forming process is required. A continuous ("production") process is necessary to get past the variations inherent in start-up to achieve uniformity. To avoid spot defects, the chemicals must be extremely clean and free of foreign particles. Making swelled film cells, therefore, requires much more stringent process control than making fluid-filled cells.

Handling swelled films is also difficult and messy, and not well suited to production, especially on non-flat surfaces. Nevertheless, it was demonstrated that curved goggle cells using swelled film can be made without defects other than the nonuniformies inherent in the film itself.

The invention of the non-swelled film represented a very significant advance in suspended particle film technology. These films are made directly in the cell itself and are much easier to handle, require no solvent, and almost eliminate spot defects due to foreign particles. Virtually perfect looking flat glass film cells were made using this type of material. Contrast of the non-swelled film is higher than that of the swelled film, although speed, to date, has been slower.

Two problems with this material, however, have so far prevented the production of good quality goggles: crazing lines and (non-uniform) loss of contrast with time. Some cells have been made which do not show much crazing, and RFI have a few cells which have survived over a year without significant loss of contrast. More work on the chemistry of the film material is required to solve these problems reliably and consistently.

It was demonstrated, nevertheless, that film cells in the form of goggles with very smooth, uniform appearance from center to edges can be made up to the stage where the film is cured by baking. After curing of the film, the above problems appear. We believe, therefore, that when these problems have been solved, it will be possible to make high quality VTVs using SP film.

We believe that the non-swelled film technology is very promising, and will eventually be capable of yielding fine quality light shutters. It is a much more recent development than the fluid suspensions, and has not yet had sufficient time to mature. Even so, it is evident that the film material has essentially solved the primary problem of agglomeration of particles in the fluids by constraining the particles in tiny droplets, or capsules isolated from each other.

The potential for making much tougher self-supporting SP films, which could be laminated onto substrates and coated with ITO, is also a future possiblity.

2.5 References

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3.0 POLYMER DISPERSED LIQUID CRYSTAL SHUTTER TECHNOLOGY

3.1 Introduction

Polymer Dispersed Liquid Crystal (PDLC) shutter technology is one of the mature, liquid crystal-related technologies. PDLC shutters were considered as viable candidates for the variable transmittance visor (VTV) application because of the following advantages over other liquid crystal shutter technologies.

a) PDLC shutters require no polarizers. These shutters are based on either scattering or absorption, therefore, no polarizers are required. The transmittance of polarizers is typically less than 50%. Hence, without polarizers the transmittance can be adjusted over a larger range.

b) They appear neutral in colour. For shutters that work in scattering mode, this is certainly the case. For shutters that work in absorption mode, they can appear neutral when proper neutral dyes are used.

c) They can be made in the form of film. Because the final shutter is in the form of a visor, the ability to made the electro-optic medium in film form would significantly simplify the fabrication of the VTV.

3.2 PDLC Materials

There are two principal types of PDLC materials. One of them was developed by Professor J.W. Doane at Kent State University, Kent, Ohio[1]. It was originally given the name "PDLC". The other type was developed by J.L. Fergason[2] at Taliq Corporation, CA. Originally this material was called Nematic Curvilinear Aligned Phase (NCAP) material. Since both types of material consist of tiny liquid crystal droplets in a polymer matrix, some people refer to both of them as PDLC materials.

Another material which was evaluated is the polymer stabilized cholesteric texture (PSCT) material, which was also developed by Professor Doane. This material is described in Section 3.6.

3.2.1 Scattering-Mode versus Absorption-Mode Operation

In a scattering-mode shutter light is blocked by scattering of the light due to the mismatch of refractive indices between the liquid crystal droplets and the polymer matrix. In the field-on state, the director of the liquid crystals nominally aligns with the external field. If the ordinary index of refraction of the liquid crystal fluid is the same as that of the binding polymer, the transmittance will be a maximum. In the field-off state, the director of the liquid

crystal is nominally perpendicular to the external field. In this case scattering is caused by both the difference between the ordinary index of refraction of the liquid crystal and the refractive index of the binder, and the difference between the extraordinary index of refraction of the liquid crystal and the refractive index of the binder. To maximize the specular scattering contrast ratio, the field-on state transmittance should be maximized, while the field-off state transmittance should be minimized. Therefore, for maximum contrast ratio, the ordinary index of refraction of the liquid crystal should be the same as the refractive index of the binder, while the difference between the extraordinary index of refraction of the liquid crystal and the refractive index of the binder should be as large as possible.

An object seen through a scattering-mode shutter appears hazy. Since there is a requirement on the maximum allowable haze in a VTV, the use of scattering mode devices should be avoided.

In an absorption-mode shutter, light transmittance is reduced by means of absorption by the dye molecules in the liquid crystal fluid. To reduce the scattering effect, low birefringent liquid crystal fluid should be used. Furthermore, the indices of the liquid crystal fluid and the polymer binder should be as nearly equal as possible. Since high order parameter, dichroic dyes are used, the dye molecules align with the liquid crystal director. In the field-off state, the orientation of the droplets (which for NCAP material is the in-plane orientation) is random. Hence the absorption axes of the droplets are randomly oriented, and the film absorbs light most effectively. In the field-on state, the liquid crystals are rotated by the external field, and the dye molecules follow. Therefore the film transmittance is higher. As will be shown below, a proper dye concentration should be used to obtain both high transmittance and high contrast ratio.

3.2.2 Doane's PDLC Material

Doane's PDLC material is prepared by phase separation. The liquid crystal fluid is first dissolved in a prepolymer. A thin liquid film of this mixture is formed, for example, by filling a thin cell. Then the liquid film is cured. As the prepolymer cures, the liquid crystal separates out from the mixture. The size of the droplets depends on the details of the curing process parameters. For example, if the prepolymer is UV epoxy, the faster the curing, the smaller the droplet size.

Doane's PDLC material is not suitable for the VTV application because it cannot operate in absorption mode. The reason is as follows. The standard way to make PDLC material for absorption mode is to dope the liquid crystal fluid with dye before it is mixed with the prepolymer. As the liquid crystal dissolves in the prepolymer, the dye molecules also dissolve. During phase separation, some of the dye molecules remain in the polymer rather than separating out with the liquid crystal droplets. The dye molecules in the polymer cannot be rotated by an external electric field. Consequently, the transmittance in the field-on state is reduced and so is the contrast ratio. Therefore, this material is not considered for use in the

VTV application.

3.2.3 NCAP Material

In NCAP material, the prepolymer is an aqueous solution of, for example, poly(vinyl alcohol) (PVA). The solubility of liquid crystal in PVA is low. Liquid crystal droplets are generated by mechanically chopping the liquid crystal fluid. If the liquid crystal fluid is dye doped, most of the dye molecules will stay within the liquid crystal fluid all the time, in contrast to the situation with Doane's PDLC material. By choosing a liquid crystal fluid with the proper refractive index, it should be possible to use NCAP material to make absorption-mode shutters. Because of this difference, more serious consideration was given to the use of NCAP material for the VTV application.

3.3 NCAP Shutter Fabrication

3.3.1 Material Preparation

In the development of NCAP shutters, PVA was the main binder material for the liquid crystal droplets, although polyethylene was also tried. 99-100% hydrolysed PVA was dissolved in warm distilled water by continuously stirring. In general, the concentration of the PVA affects the size and shape of the droplets, and also the resultant film thickness. The liquid crystal droplets were generated by a mechanical emulsifier. Hence the higher the PVA concentration and the more viscous the solution, the more difficult it was for the emulsifier to produce small droplets. Because the film was made by evaporating the water, the original spherical droplets became elongated as the film shrank. The greater the shrinkage of the film, the more elongated the droplets became. It was found that 10 - 15% PVA solution produced satisfactory NCAP films.

In shutters designed to work in absorption mode, the liquid crystal fluid is doped with the proper amounts of dyes. Only high order parameter dichroic dyes were used in our work. Sometimes heating was required to dissolve the dyes completely in the liquid crystal fluid. The mixture was then poured into the PVA solution and emulsified (broken into droplets and dispersed throughout the PVA) by a mechanical emulsifier. The size of the liquid crystal droplets generated by this process depends on the viscosity of the mixture, the spinning speed of the emulsifier, the duration of mixing, and the temperature of the mixture during emulsification and before complete polymerization of the PVA binder. The duration of the emulsification process required to generate small droplets depends on the amount of the mixture. For a large volume of mixture, the spinning time required was so long that the mixture was covered to reduce the evaporation of the water. The size of the droplets was monitored during the emulsification. The droplet size used in our shutters was about 3 μm in diameter.

An important and difficult problem in the fabrication of NCAP films is that of degassing the mixture. Because the liquid crystal fluid was emulsified by vigorous mixing of the two materials in air, a large number of tiny air bubbles always became trapped in the mixture. Attempting to degas the mixture in a vacuum chamber resulted in rapid evaporation of water, particularly from the exposed surface of the mixture. The situation was greatly improved by establishing a water vapour-saturated environment inside the vacuum chamber during degassing, as described in Section 2.2.3. However, this process was not completely effective. A layer of dryer material usually formed on top of the mixture during the degassing process. The resulting inhomogeneities in the mixture interfered with the production of smooth films. To prevent this problem, only the material underneath the top surface is used to make films.

3.3.2 Film Fabrication

In the early stage of the development, NCAP films were made by spreading the emulsified mixture over a clean piece of glass with a razor blade, and then allowing the film to dry in air. This method can only be used to make small size films, say squares less than 2"x2". The spreading motion must be very uniform to avoid the formation of ridges in the film.

Subsequently films were made by spin coating and then drying in air. By this method smooth NCAP films could be made quite easily, provided the mixture was homogeneous, and was spread in a dust-free environment. Any particles that fall on the wet film form tiny circular spots. The film thickness could be easily controlled by the spin speed. The size of the films could be made quite large without encountering any thickness uniformity problems.

While spin coating on a flat surface is quite easy, it is not trivial to spin coat a smooth film on a curved surface. Some spin coating experiments were conducted on the inner surface of a visor. The visor was held by a vacuum spinning chunk with a thick O-ring on top. The thick O-ring provided an extension of the vacuum chunk. It was realized that good control of both the initial low spreading speed, and the final high speed spin was necessary to obtain films without a small central circle. Further work is clearly necessary to achieve uniform, high quality films on curved visor surfaces.

3.3.3 Shutter Fabrication

To make planar shutters the NCAP material was first spun onto an ITO-coated glass substrate. After the film was completely dried, a second ITO-coated glass substrate was placed on top of the NCAP film. This sandwich was then pressed inside a vacuum bag. If there were gaps between the film and the second substrate, the vacuum bag was placed inside a 60°C oven for about 30 min. This process brought the film into contact with both

substrates. Finally the NCAP material outside the cell was removed and the edges were sealed with epoxy. The thickness of the NCAP films was typically 7-10 µm. NCAP sample shutters of various areas were fabricated in this way. The largest shutters made were about 8"x 8".

3.4 Optical Measurements
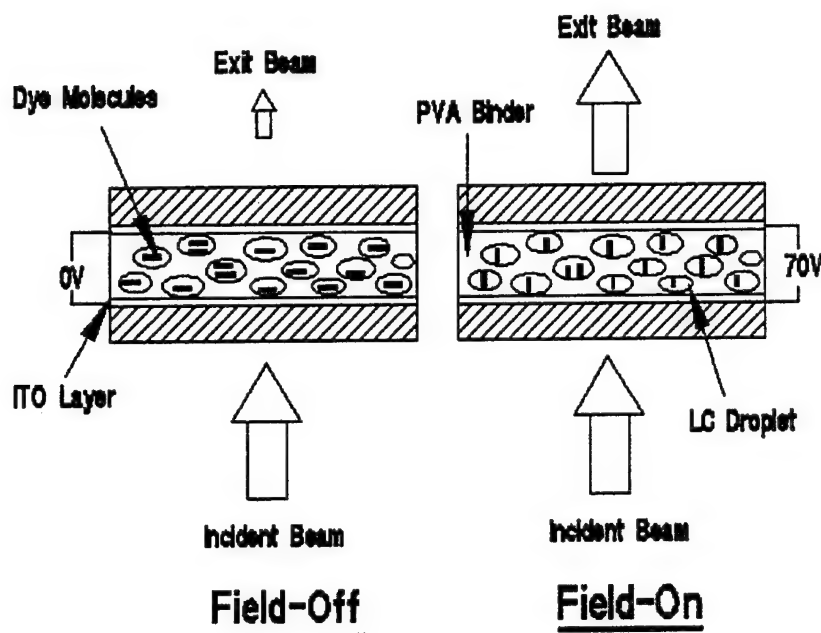
3.4.1 Shutter Characterization

Figure 3-1. Schematic diagram of an NCAP shutter.

Figure 3-1 shows a schematic diagram of the cross section of an NCAP shutter in the field-off and field-on states. In the field-off state, as explained above, the dye molecules are oriented randomly in the plane of the shutter. Light with incident directions sufficiently close to the normal to the shutter will be partially absorbed by the dyes. When an external field is applied, the liquid crystals are rotated to line up with the external electric field. Because the dyes have high order parameter, they are also aligned to the external field, and permit most of the light to be transmitted. If the liquid crystal fluid has a very low birefringence and a refractive index nearly equal to that of the PVA, there should be no scattering in either the field-on or field-off states.

The index of refraction of PVA is 1.526. The best available liquid crystal mixture for an absorption mode NCAP shutter is ZLI1800-000 which has $\Delta n = 0.078$, and $n_e = 1.556$. Even with this liquid crystal mixture, the scattering in the resulting shutter is still not

negligible.

The optical performance of a PDLC shutter can be characterized by its specular transmittance, specular contrast ratio, and percent haze. For the VTV application, sufficiently high specular transmittance, high specular contrast ratio and low haze are required.

Percent haze is generally defined as

$$\%Haze = \frac{100\ T_s}{T_t}\ ,\tag{3.1}$$

where T_t is the total transmittance and T_s is the scattered transmittance. However, to properly correlate it with the measured quantity, the percent haze should be defined as[3]

$$\%Haze = 1 - \frac{T_{f/7}}{T_{f/0}}\ ,\tag{3.2}$$

where $T_{f/0}$ is the total transmittance and $T_{f/7}$ is the specular transmittance measured by a detector with f/# = 7 optics[3], i.e. referring to Figure 3-2,

$$\frac{D}{d} = 7\ .\tag{3.3}$$

If the scattering of the optical instruments is zero, the percent haze defined in Equation 3.2 is the same as that used in ASTM D1003-61(R88). Specular contrast ratio at voltage V is then defined as

$$CR(V_{applied}) = \frac{T_{f/7}(V = V_{applied})}{T_{f/7}(V = 0)}\tag{3.4}$$

3.4.2 Optical Setup

Figure 3-2 depicts the optical setup used to measure the specular transmittance, specular contrast ratio and percent haze. Lens L1 collimates the light from the tungsten light source S1. The beam size of the collimated light is reduced by a diaphragm PD. An integrating sphere IS is placed in either of the two locations, D = 0 and D = 7 d. In location D = 0 $T_{f/0}$ is measured, and in the other location $T_{f/7}$ is measured. A light blue filter F1 is placed in front of the input opening of the integrating sphere to reduce the infrared from the light source. A silicon pin diode detector DI is mounted on top of the integrating sphere. The output of the pin diode is sent to a current-to-frequency converter. The frequency is then recorded by a computer. The computer also controls a power supply connected to the NCAP shutter. A computer program was written to vary the applied voltage and record the light

intensity in the pin diode. The applied voltage is a square wave varied from 0 V to about 70 V, in steps of about 2V. There is a waiting period of 0.5 sec between successive applied voltages. All the measurements were done along the normal to the shutters. The typical error in the percent haze measured by this setup is better than ±2.
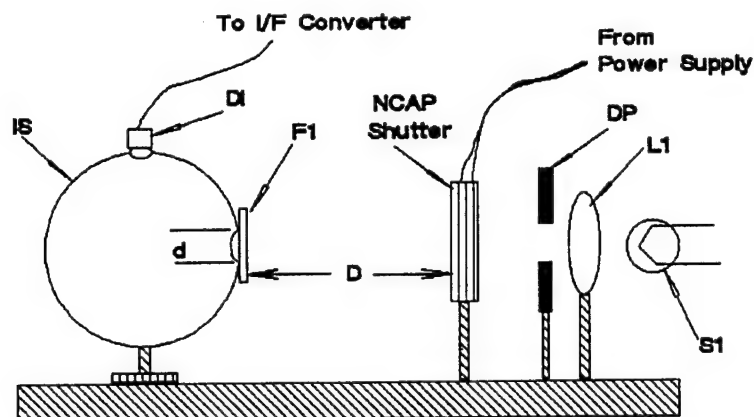

Figure 3-2. Setup for optical measurement.

3.4.3 Results and Discussion

The following three figures demonstrate the effect of dye doping and birefringence on specular contrast ratio and percent haze in NCAP shutters. The optical parameters, at room temperature, of the liquid crystal fluids and the PVA used in this study are listed in Table 3-1

Table 3-1. Optical parameters of liquid crystal fluids and PVA.

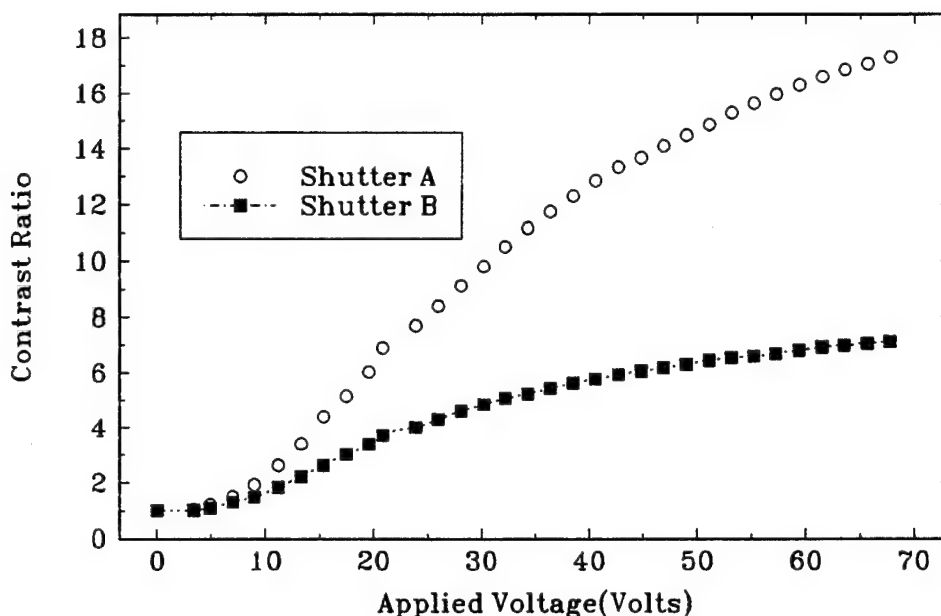|	n_0	n_e	Δn
PVA	1.526	N/P	N/P
E83	1.5225	1.761	0.238
ZLI4284	1.486	1.5845	0.0988
ZLI1800-000	1.488	1.556	0.078

for reference.

Figure 3-3. Effect of dye doping on specular contrast ratio.

Figure 3-3 shows the effect of dye doping on the specular contrast ratio. The host liquid crystal fluid in both shutters is E43. However, Shutter A is doped with dichroic dyes, while Shutter B is not. Other film parameters are the same. At the maximum operating voltage, the dye-doped shutter has a contrast ratio more than double that of the non-doped shutter. The contrast ratio in Shutter B is due to scattering only, while in Shutter A both scattering and absorption contribute to the contrast ratio.

Figure 3-4 shows the effect of birefringence on the specular contrast ratio. The dye concentrations in both Shutters A and C are the same. However, Shutter A contains host liquid crystal E43, which has a birefringence of 0.238, while Shutter C contains ZLI4284 as its host liquid crystal, having a birefringence of 0.0988. Since the dye concentrations and other film parameters in both shutters are the same, the difference in their specular contrast ratios can be attributed to the difference in the scattering cross sections, which is caused by the different birefringences of their host liquid crystal fluids. Furthermore, the fact that the specular contrast ratio of Shutter A is larger than that of Shutter C is consistent with the mechanism described above.
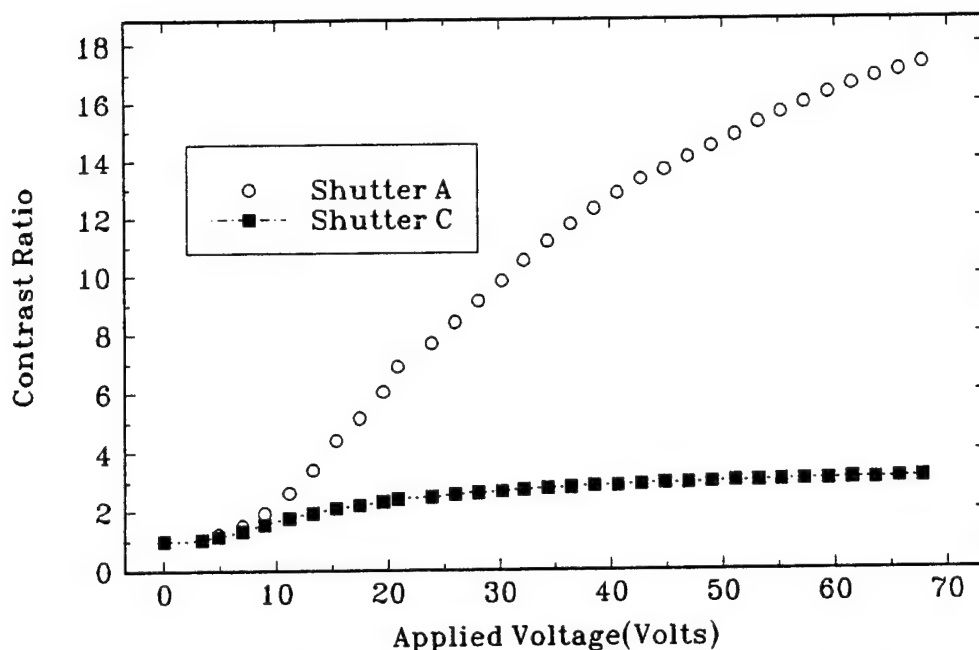
Figure 3-4. Effect of birefringence on specular contrast ratio.

Figure 3-5 shows the effect of birefringence on the percent haze. These measurements were also made with Shutter A and C. In the field-off state the percent haze of Shutter A is larger than that of Shutter C because E43 has a larger birefringence. As the applied voltage increases, the difference in percent haze between these two shutters decreases. At the maximum applied voltage both shutters reach about the same percent haze of 17.

Based on the above scattering mechanism and on the ordinary indices of refraction of the two liquid crystal fluids and of the PVA , one would expect that the percent haze in Shutter A would be smaller than that of Shutter C at very large applied voltages. The fact that they decrease to about the same percent haze suggests that there are other sources of

Table 3-2. Film parameters of shutters.

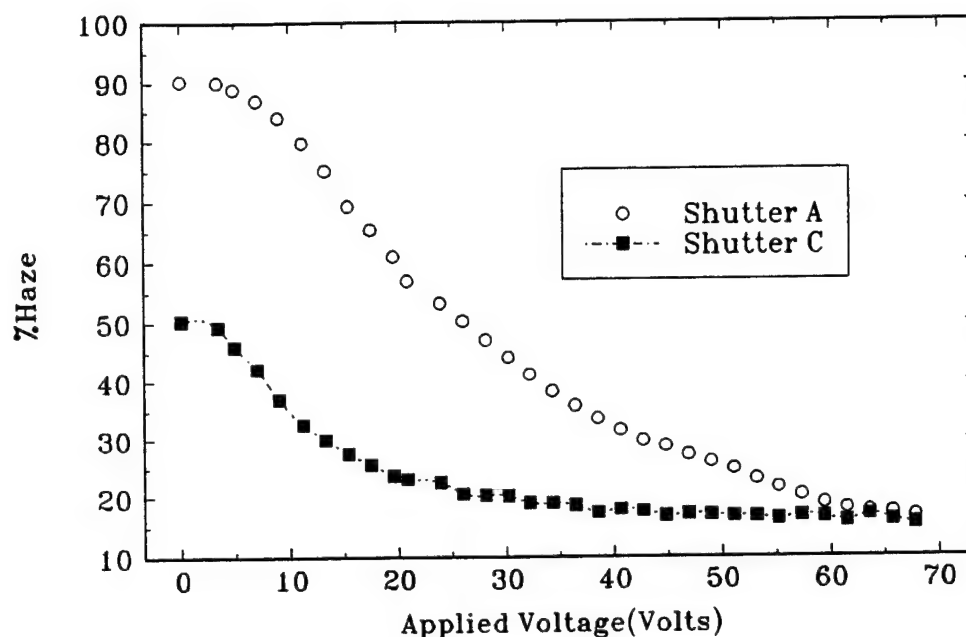| Shutter	D	E	F	G	H	I	J	K	L	M
Symbol	●	○	■	□	▲	▼	△	▽	◆	◊
Dye %	2	2	2	4	4	4	6	6	6	6
Spin Speed (x10 rpm)	125	150	175	100	125	150	125	150	175	200

Figure 3-5. Effect of birefringence on percent haze.

scattering present than simply refractive index mismatch. Possible sources of scattering include randomness in droplet size distribution and spatial distribution, and variations in droplet transmittance due to differences in the alignments of dye molecules.

Figure 3-6 shows a comparison of the specular contrast ratio and the percent haze at different applied voltages of a shutter having dye-doped ZLI1800-000. The specular contrast ratio increases with applied voltage, while the percent haze decreases with applied voltage.

The following three sets of measurements show how the dye concentration and film thickness determine the specular transmittance, specular contrast ratio and percent haze. With these data, one can have some idea what the shutter film parameters should be for a given allowable percent haze and required specular contrast ratio. This study used ZLI1800-000 as the host liquid crystal fluid because its birefringence is quite low while its index of refraction is close to that of the PVA. Table 3-2 shows the film parameters of the ten shutters used in this study. The higher the spin speed, the thicker the film.

Figures 3.7 shows the specular transmittance of these ten NCAP shutters (D to M). The specular transmittance increases with applied voltage, and decreases with film thickness and dye concentration. Figure 3-8 shows the specular contrast ratio of these shutters. The general trend is the thicker the film and the higher the dye concentration, the larger the variation in the contrast ratio as the applied voltage increases. Figure 3-9 shows the percent haze of these
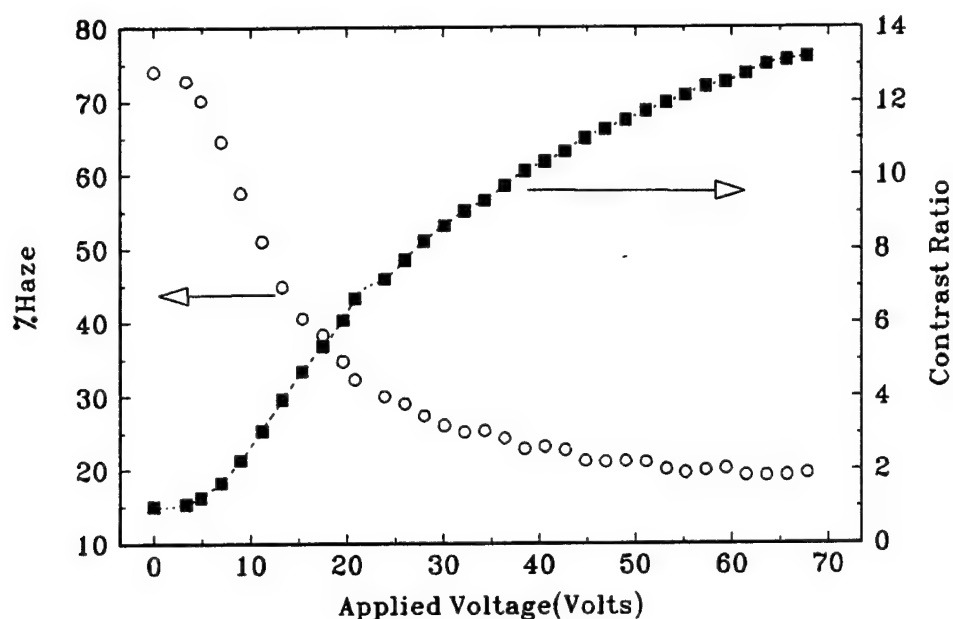
Figure 3-6. Comparison of specular contrast ratio and haze in a single shutter.

shutters. It seems that in general, the thicker the film and the higher the dye concentration, the higher the percent haze. Some of the cross-over between the data from different shutters might be due to experimental error.

3.5 Principal Problem Areas in NCAP Shutters

The principal problem areas encountered in making NCAP light shutters suitable for the VTV application are summarized below, and possible improvements which could be made are also discussed.

a)   Haze. The primary problem encountered with all of the NCAP films investigated is the magnitude of the haze. Even in films using ZLI1800-000 as the host fluid, the haze is higher than that acceptable for use in a VTV. As shown in Figures 3-8 and 3-9, films having a contrast ratio of only 5 have percent haze values in the range of 20 to 45 (Shutter L). It is entirely possible, however, that the present haze levels can be significantly reduced by further optimization of the refractive indices. However, as mentioned above, besides the refractive indices, the sizes of the droplets and the randomness of the droplet absorption might contribute some haze.

b)   Degassing. Removing trapped air from the PVA/liquid crystal/dye mixture, without
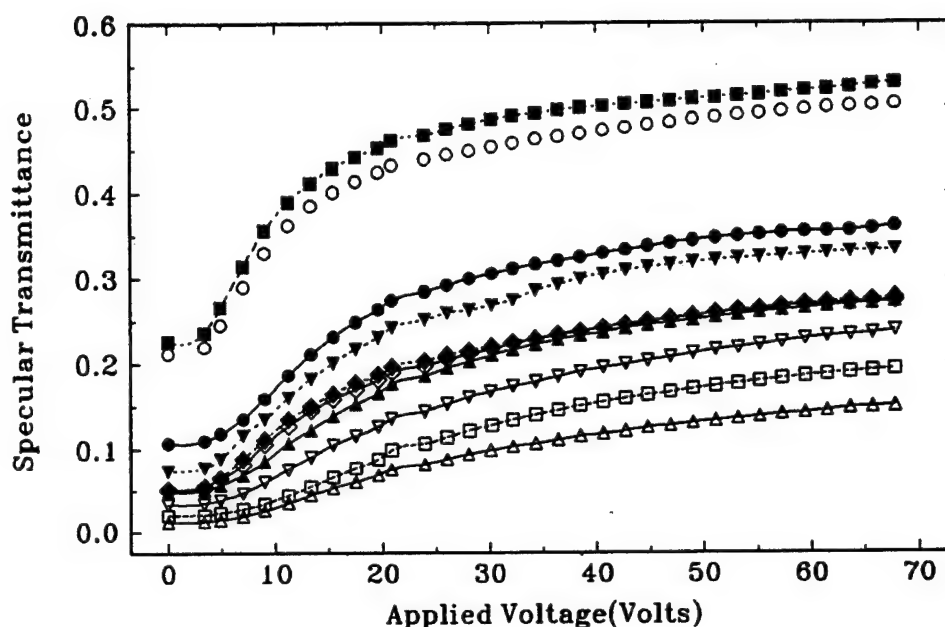
Figure 3.7. Effect of dye concentration and film thickness on specular transmittance.

simultaneously losing water, cannot be achieved in a simple manner by conventional vacuum degassing. Although the situation is much better when the degassing is done in a water-saturated chamber, drying of the top layer of material still occurs. Furthermore, such a degassing method might not be good enough if further processing of the film is required in higher vacuum. Another possibility is to mix the PVA and liquid crystal in a different environment, say in a helium atmosphere, so that subsequent degassing would require less pumping. Another alternative might be to centrifuge the bubbles out of the mixture. Centrifuging, however, will not remove dissolved air.

c)   Spin Coating on Curved Surfaces. Achieving a uniform film by spin coating on a curved surface is more difficult than achieving a uniform film on a flat surface, and it may not even be possible if the curvature of the surface is too large. Better spinning equipment is necessary in order to further investigate this problem.

d)   ITO Deposition. A desirable configuration for a VTV is an ordinary visor deposited with a layer of ITO, followed by a light shutter film (such as an NCAP film), followed, in turn, by the second ITO layer plus a hard protective coating. There would be no second substrate. Such a configuration, of course, requires the deposition of ITO on top of curved surfaces, namely the visor and the NCAP film. Experiments to investigate the deposition of ITO on planar NCAP films gave inadequate results. The resultant shutter required a much higher operating voltage, and the ITO layer was quite yellowish colouring. ITO deposition
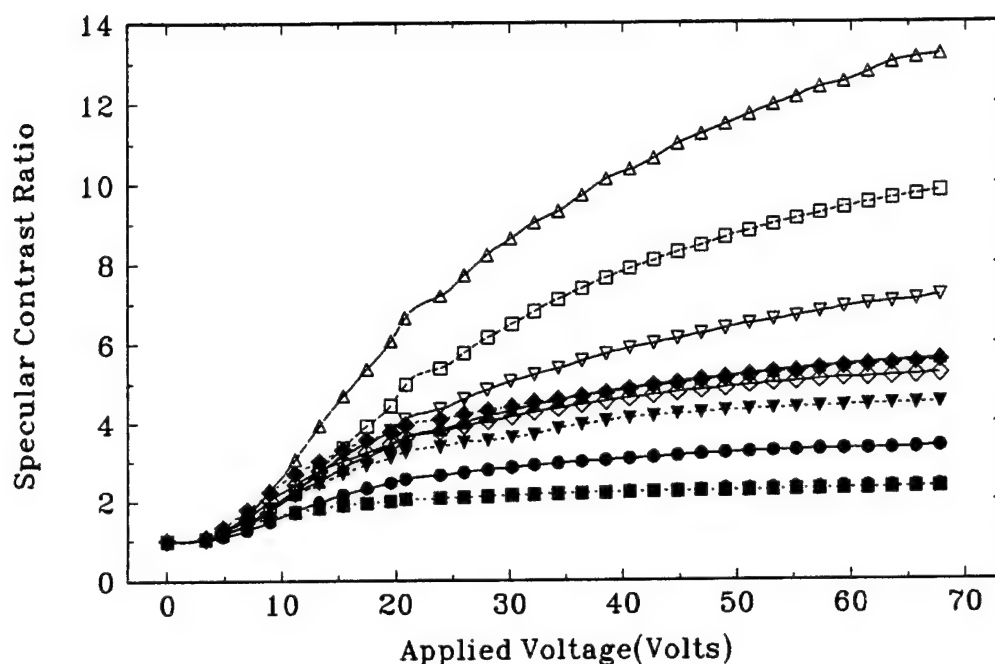
Figure 3-8.Effect of dye concentration and film thickness on specular contrast ratio.

must be done in high vacuum. The higher operating voltage might be due to the increased film thickness because of the expansion of the residual air in the dried film. The yellow color of the ITO layer might be due to the oxygen concentration used in the deposition. Additional work is required in this area.

e)   Droplet Size. As mentioned in the discussion of Figure 3-5, some of the haze might be caused by random variations in droplet properties such as size and absorption. Reducing the average droplet size might have a positive impact on the haze problem.
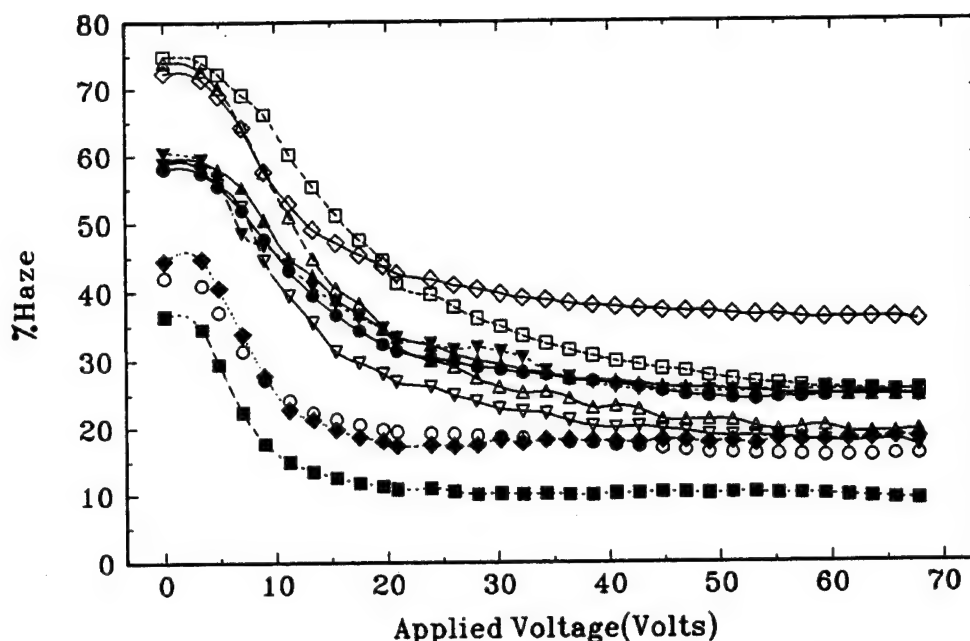
Figure 3-9. Effect of dye concentration and film thickness on haze.

3.6 Polymer Stabilized Cholesteric Texture Shutters

3.6.1 Basic Properties of Polymer Stabilized Cholesteric Texture (PSCT) Material

PSCT material[4] can be considered to be another version of PDLC material. A PSCT shutter contains a minute amount of polymer material dispersed throughout a cholesteric liquid crystal fluid. Cholesteric liquid crystal fluid exhibits three types of texture: planar, focal conic and homeotropic. Without the polymer the cholesteric liquid crystal fluid in a cell will stabilize to a planar texture. The polymer material causes the liquid crystal fluid to form domains and can stabilize the focal conic texture in the field-on or field-off state depending on the preparation procedure. PSCT shutters can be made to operate in two modes; normal mode and reverse mode. In normal-mode operation, the focal conic texture is stabilized in the field-off state. Therefore the cell scatters light in the field-off state and is clear in the field-on state. In reverse-mode operation, the liquid crystal fluid has planar texture in the field-off state and is non-scattering. In the field-on state, the liquid crystal fluid is driven to focal conic texture and therefore scatters light. PSCT material can also be doped with dye molecules. The main advantages of this kind of shutter are :

a) Low haze at all viewing directions in the field-on state of a normal-mode shutter; and

b) Choice of normal- or reverse-mode operation.

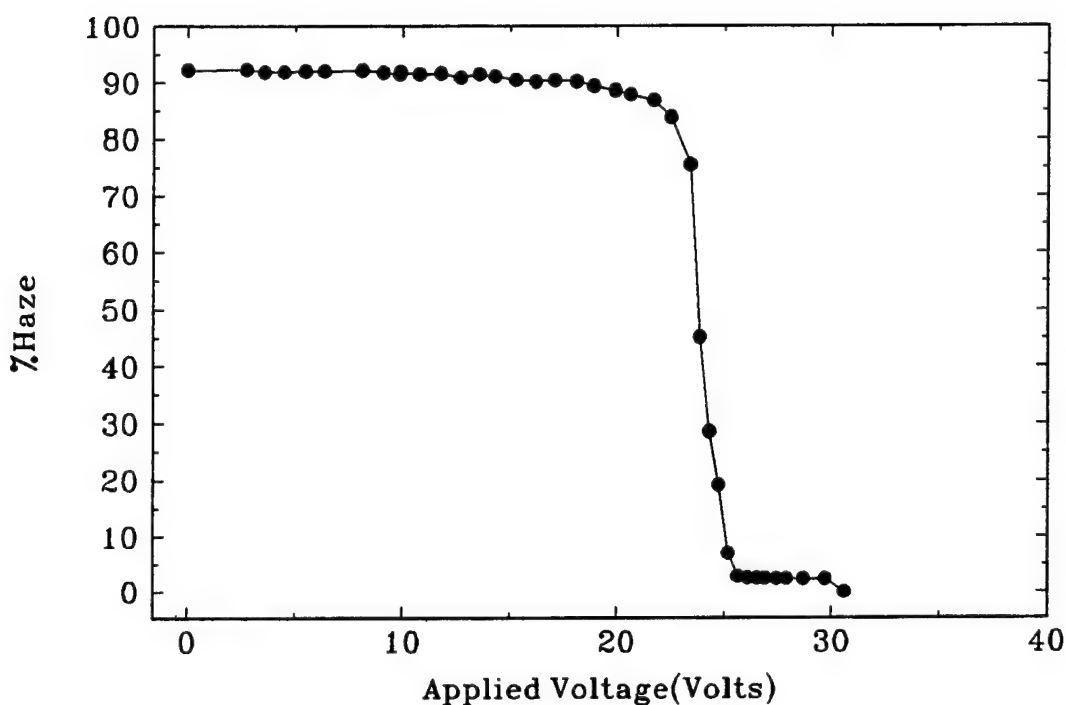### 3.6.2 Percent Haze Characteristics

Figure 3-10. Percent haze in a PSCT shutter sample.

A PSCT shutter was obtained from Kent State University. It was a dye-doped normal-mode shutter. The liquid crystal fluid in this shutter has a birefringence of about 0.2. Figure 3-10 shows the percent haze of this shutter. In the field-off state, the percent haze was larger than 90. In the fully field-on state, the percent haze was less than 3. It was observed that the density of spacers in this shutter is high. Therefore, some of the haze could be due to light scattering by the spacers. In the fully field-on state, there is no apparent haze difference in this shutter when it was viewed at different viewing directions.

The main source of light scattering in the field-off state was the polymer-stabilized focal conic texture. The scattering from the focal conic texture should be less if a host liquid crystal fluid with a low birefringence is used. However, based on our measurements on

White-Taylor dichroic liquid crystal shutters, even using low birefringence liquid crystal fluids such as ZLI1800-000, the percent haze caused by the focal conic texture was non-negligible. It is not clear at this point whether or not there is a liquid crystal fluid available to fabricate a non-scattering absorption-mode PSCT shutter. If no suitable material can be found, then this technology is not suitable for the VTV application.

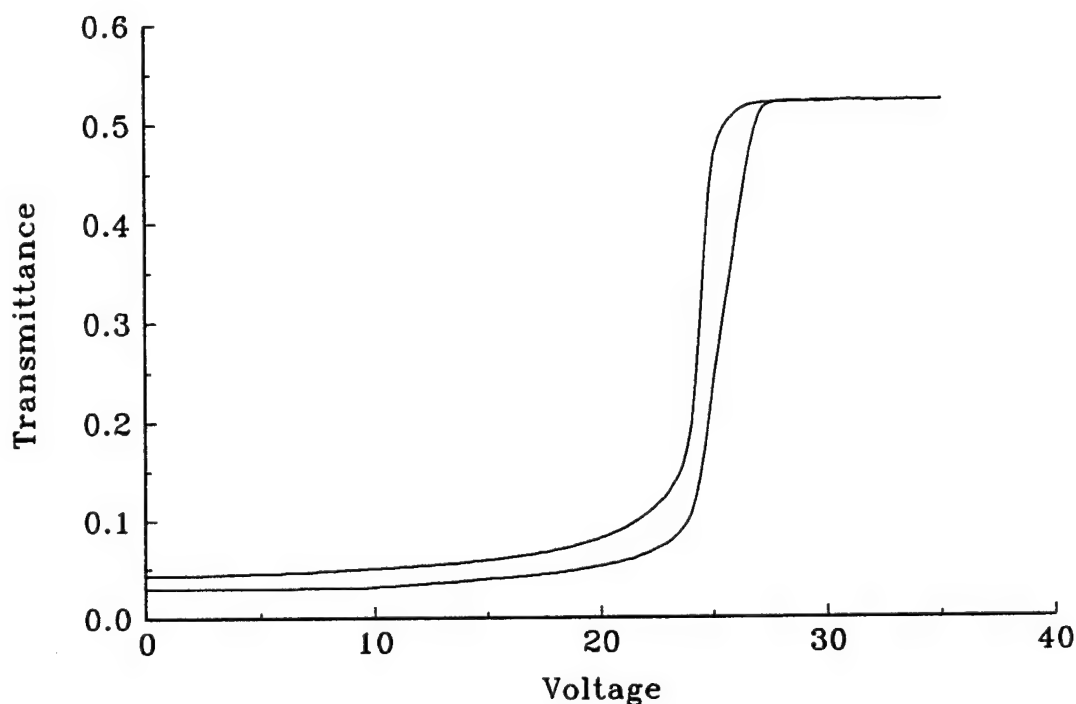### 3.6.3 Threshold Characteristics

Figure 3-11. Threshold characteristic of the PSCT sample.

The quasi-static threshold characteristic of the sample shutter is shown in Figure 3-11. The lower curve is the ascending branch and the upper the descending. The saturated contrast ratio on the ascending branch is 17.8 and on the descending branch is 12.1. The voltage difference at the 50% point is 0.365 V. Because the phase changes take a finite time, the details of the transition regions are a function of voltage sweep rate. Once the cell is operating on the upper branch it stays there, at least for some tens of minutes, until the drive voltage is "instantaneously" reduced from above to below the transition region.

Despite the combination of the hysteresis and the large rate of change of transmittance with voltage in the transition regions, use of the feed back control system described in section

4.2 should provide satisfactory operation.

3.7 Conclusions

Initially, NCAP material appeared to have a good potential for use in the VTV application. This is because of its high transmittance, ease of being formed into film on curved surfaces and its use of a well established electro-optic material, namely, liquid crystal fluid. The technology is fairly mature, at least for planar shutters.

However, our investigations led to the recognition of a number of material and fabrication problems which currently prevent the production of a practical VTV using NCAP material. Although we believe that these problems can be solved, much more work would be required.

An evaluation of the potential of using PSCT for the VTV application has been performed. It was found that, except in the fully on state, this type of shutter has a large haze problem. Without substantial improvement in this technology it would not be suitable for use in VTVs.

3.8 References

1. Doane J.W,. Vaz N.A., Wa B.G., and Zumer S., Appl. Phys. Lett. 48, p269.

2. Fergason J.L., US Patent 4,435,047.

3. Drzaic, Paul S., Mol. Cryst. Liq. Cryst., Vol. 198, pp. 61-71 (1991).

4. Doane, J.W., D.K. Yang and L.C. Chien, Conf. Rec. IDRC, SID, p.175 (1991).

4.0 CONTROL SYSTEM

4.1 Introduction

The VTV must be provided with a control system that is capable of operating in either of two modes: a constant transmittance mode or a constant luminance mode. In general, the designer of the control system can expect that the voltage to luminance transfer characteristic of the visor will be nonlinear, temperature dependent, and slightly variable from unit to unit. He can therefore provide the control unit with complementary nonlinearity, temperature dependence and adjust-on-test capability or he can employ negative feedback techniques. Properly implemented, the latter approach can significantly simplify the control system.

4.2 Luminance Control System

A simple proof of concept constant luminance control system using negative feedback was developed and tested. This took the form of a luminance regulating servo as shown in Figure 4-1.
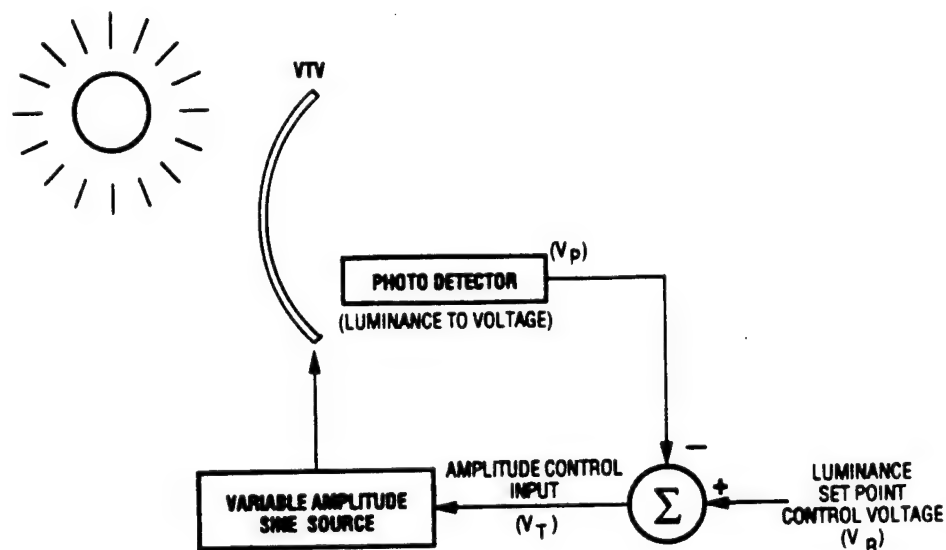

Figure 4-1. Luminance control system.

The luminance inside the visor, L_O, was converted to a proportional voltage, V_P, using a photo diode with an approximately photopic response and a transresistance stabilized amplifier. The transducer constant is denoted K_P. The output voltage of the amplifier was subtracted from a luminance set point control voltage, V_R, and the difference voltage, V_T, controlled the transmittance of the visor. The visor was a TN shutter with a contrast ratio of 50:1. Its voltage to transmittance transfer function is shown in Figure 4-2. The shutter was driven with a 1000 Hz sine wave from a function generator with an amplitude modulation capability. The control voltage V_T was connected to the amplitude control input. In order to simplify the analysis it is assumed that the visor transmittance is proportional to the control voltage and the transducer
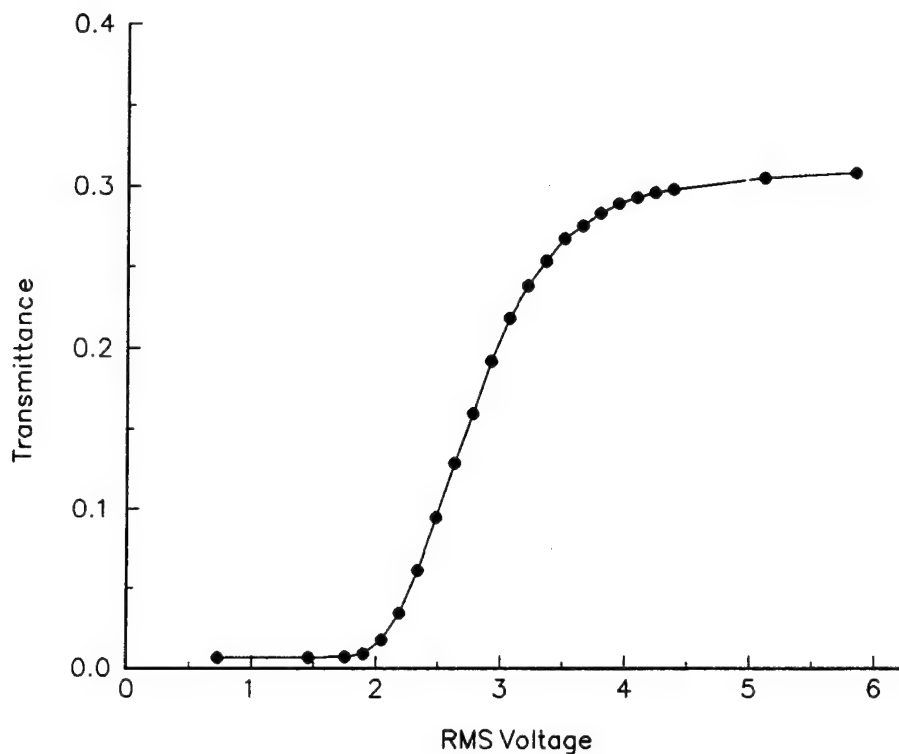
constant is denoted K_T.

Figure 4-2. TN shutter voltage transfer characteristic.

Denote the luminance into the visor as L_I. A little algebra shows that the relationship between L_O and L_I is:

$$L_O = \frac{V_R}{K_P\left[1+\dfrac{1}{L_I K_P K_T}\right]}$$

If K_P and K_T are selected such that $L_I K_P K_T \gg 1$ then

$$L_O \simeq \frac{V_R}{K_P}$$

The schematic of the breadboard that was built to test the servo concept is shown in Figure 4-3. It is straightforward except for the two diodes in the inverting amplifier. These prevent the amplitude modulation input of the function generator from swinging negative. The output amplitude of the function generator is independent of the modulation input polarity. Thus if the modulation signal is allowed to reverse polarity the system can lock up if the input luminance drops very rapidly.
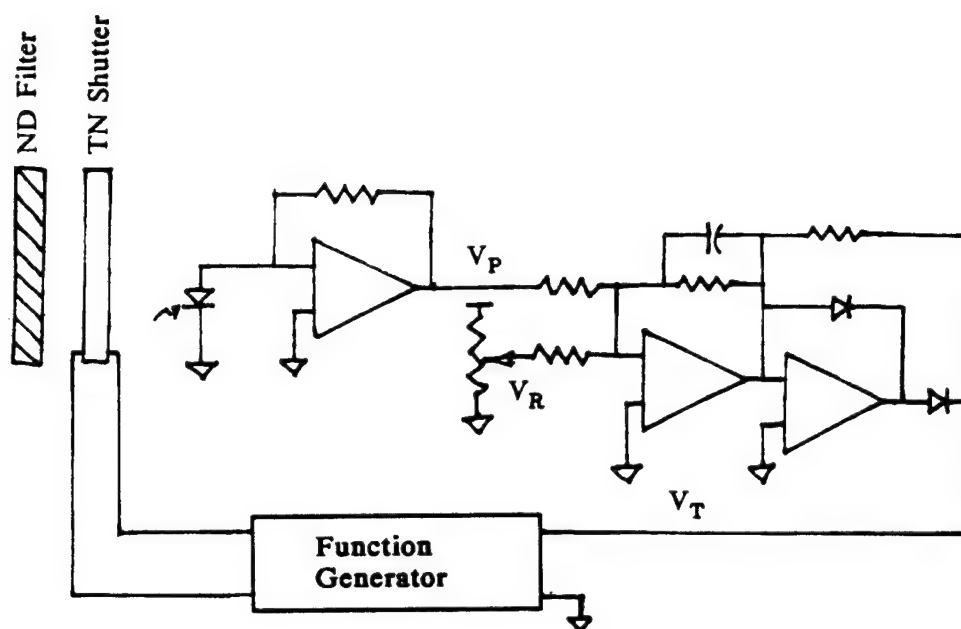
Figure 4-3. Luminence control breadboard.

The values for the constants in the breadboard system are:

K_P = 0.0834 V·(luminance units)$^{-1}$
K_T = 10.0 V^{-1}
V_R = 0.414 V
$\dfrac{V_R}{K_P}$ = 4.96 luminance units

The performance of the breadboard was evaluated by inserting neutral density filters in front of the shutter. The results, plotted in Figure 4-4, are as follows:

L_I	L_O
1000	5.28
500	5.00
250	4.96
100	4.90
50	4.86
25	4.74
12.5	4.14
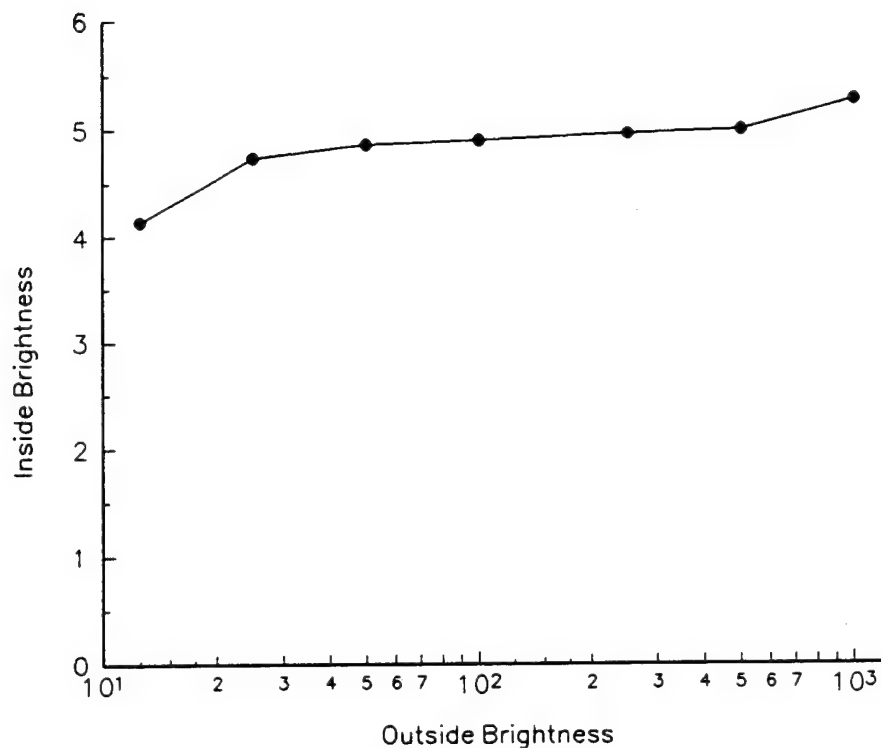
Figure 4-4. Breadboard performance.

This is as expected. Over the input range of 50 to 500 units the output luminance is almost contant. Outside this range the control degrades due to saturation in the shutter. The input luminance range is 80, while the contrast ratio of the shutter is only 50.

4.3 Conclusions

The luminance feedback control system tested is a good method of accomplishing the constant luminance control function. It proved to overload gracefully and, with proper design, to exhibit no undesireable modes of operation. We believe it to be a viable control technique for application to the VTV.

5.0 CONCLUSIONS

Three principal areas were investigated during this project: Suspended Particle technology, Polymer Dispersed Liquid Crystal technology, and electronic control to regulate visor transmittance.

The main concentration of effort on shutter technology for the visor was directed towards the development of SP shutters because this technology appeared to offer the greatest promise of success in the shortest time. Fluid-filled SP cells were already being made before the commencement of this project, and it was clear that the technology did work.

Nevertheless, a number of deficiencies were recognized with respect to the performance characteristics of the SP materials. These deficiencies included a) inadequate stability of the suspensions, which resulted in agglomeration of the suspended particles into visible spots and streaks, b) significant loss of speed, which generally accompanied attempts to increase stability against agglomeration (because of increases in viscosity), c) insufficient contrast within the desired transmittance range, d) less than adequate resistance to high temperatures, e) higher operating voltages than desired, and f) blue colour.

A further recognized disadvantage of SP materials is that they could not be simply coated onto the visor surface. They could operate only when enclosed in cells with two walls.

It was believed that an understanding of how to solve most of these problems was at hand and that their solution would probably be achieved within the time frame of the project.

Over the duration of the project, significant advances were made in the SP fluids through the use of new types of crystals and polymers. These developments resulted in improved electrooptical performance (particularly higher contrast and lower operating voltages for the same cell gap), and greatly improved resistance to high temperatures. As a result of these improvements, the required VTV transmittance range of 7% to 70% was met, operating voltage in the range of 20 volts was achieved, and resistance to temperatures well in excess of the upper limit of the military temperature range was achieved (up to 130°C).

However, eliminating agglomeration of particles in fluid shutters, while simultaneously retaining fast switching times, high contrast, and low operating voltages, proved to be much more difficult than anticipated. Many test cells were made which functioned well and were generally uniform in appearance except for visual defects which either appeared immediately after filling the cells, or a few days or weeks later. These defects took the form of dark spots and streaks, or even larger zones and bands. These problems were not satisfactorily resolved before the end of the project, and this prevented the fabrication of SP fluid-type VTVs of acceptable quality within this time frame.

Substantial progress was made in the technology as a result of the invention and development of Polymer Dispersed Suspended Particle (PDSP) films, particularly the non-swelled type. These films are virtually free of the problems of agglomeration exhibited by the fluid materials. SP films also have advantages over SP fluids in that they are not subject to settling due to specific gravity imbalances, they hold the cell substrates together over their entire area, and there can be no fluid flow within the cells (and hence no transient visible effects due to pressure being applied to the cells).

Virtually perfect-looking 4" x 7" flat glass test cells were made using the most advanced available film materials (which were usually synthesized at RFI in experimental quantities, and were always in short supply). Shutters in the form of goggles were also made (using mating pairs of individual goggles), with the intention that these would serve as prototype VTVs for demonstration and test purposes. These goggles looked virtually perfect initially, thereby demonstrating that curved SP shutters can be made using polycarbonate visors.

However, almost all of the test cells, including the goggles, that were made using the new PDSP films suffered substantial degradation either shortly after final cure in the oven, or some days or weeks after that. The problems included a) the appearance of fern-shaped patterns of dark lines (referred to as "crazing lines") mainly around the edges of the cells after the film was oven-cured, and b) loss of cell contrast, usually in a non-uniform pattern within the cell area. Some success was achieved in reducing both of these problems near the end of the project, but, again, not sufficiently to make possible the production of PDSP goggles having sufficiently good quality to serve as prototype VTVs for demonstration and test purposes. Another problem with the new non-swelled films, unlike the fluids and swelled type films, is that their speed was slow.

Significant progress was also made in the processes for fabricating SP cells of various types. Fluid cells were filled using a vacuum fill process. Procedures for handling the fluids to permit degassing, while preventing the loss of volatile materials from the fluids under vacuum conditions were developed. Procedures were also developed to assemble PDSP film cells under high vacuum to prevent the entrapment of air. These procedures made possible the assembly of the prototype goggle VTVs.

One other difficulty with SP materials, which has not been resolved, is its blue colour. To solve this problem permanently requires the development of different types of suspended particles with different spectral absorption characterisitics. The problem could be partially alleviated in the short-term with the addition of a compensating colour filter. This would not be a complete or final solution, however, because uniform colour compensation cannot be achieved over the full range of shutter transmittance with a single filter. This is because the spectral transmittance characteristic of the shutter changes as the overall transmittance changes (i.e. as the operating voltage is changed).

As a result of this project, it has been demonstrated that SP materials are capable of meeting the VTV requirements for contrast (including both minimum and maximum transmittances), temperature, and operating speed. It has also been demonstrated that visors, employing specially designed, two-piece mating pairs, can be made using this type of material. The main obstacle to the production of acceptable VTVs using SP technology at this time is inadequate stability of these materials. The primary problem of agglomeration of particles in the fluids has been essentially solved already by the development of the PDSP films. We believe, therefore, that when the remaining difficulties with the non-swelled films have been solved, it will be possible to make VTVs using SP films.

Substantial effort has been put into the development of NCAP material for use in the VTV application. Basic properties of NCAP shutters, such as the dependence of specular transmittance, specular contrast ratio, and percent haze on dye concentration, refractive index of the host liquid crystal fluid, refractive index of the PVA, birefringence of the liquid crystal fluid, film thickness, and droplet sizes have all been studied. NCAP shutters of various sizes were made by spreading the film using either a razor blade or a spin coater.

During these investigations, it was determined that the percent haze could be greatly reduced at low applied voltages by changing the host liquid crystal fluid from E43 to

ZLI1800-000. It was shown that, with sufficient dye concentration, the dye-doped NCAP shutters are capable of contrast ratios of greater than 12:1. It was also demonstrated that electrooptically active material can be coated directly onto the inner surface of a visor. This represents a significant step toward the development of a simple VTV fabrication process. Some degree of success was also achieved in coating conducting ITO layers onto NCAP films directly. Functioning shutters made in this manner, on a single substrate, were produced, although the contrast obtained was low.

A variety of problems were identified during the course of this work. Some of these problems, such as achieving proper degassing of the film material and laying down high quality films on substrates, were partially solved. This was evident through the succession of improvements which were obtained in the films produced throughout the course of the project, even though films of sufficiently high quality for use in a VTV were not achieved.

Other problems, however, are more fundamental and will likely require greater advances in the technology before they can be solved. These problems include the generation of fairly uniform droplet sizes much less than 3 μm, and producing host liquid crystal fluid having the proper refractive index with respect to that of the PVA.

A Polymer Stabilized Polymeric Texture (PSCT) shutter was examined to evaluate the feasibility of using this technology for VTVs. At this time it appears doubtful that PSCT technology would be a suitable candidate for VTVs in the near future because of the very large haze levels observed. To solve this problem would require a new liquid crystal material which, to the best of our knowledge, is not currently available.

A prototype feedback control system to maintain the pilot-set see-through luminance level constant was designed and tested by building a breadboard model. The system operated successfully. It has, therefore, been demonstrated that the automatic luminance control function of the VTV can be achieved using a circuit of this type, and no significant problems are anticipated in this area.

To make a projection of the time and resources required to solve the remaining problems with the SP films involves a number of considerations. These can be approached from two different perspectives: 1) what is the likely rate of future progress of work on SP films in relation to past progress, and 2) what understanding presently exists of the nature of the problems to be solved?

On the positive side, improvements have been completed just recently in the research facilities at RFL. A new analytical chemistry laboratory has been opened, which will allow more modern processes and procedures to be employed. For example, the addition of a chromatograph will make possible the accurate determination of molecular weights of the polymers, which is important in developing materials with particular desired properties. It is expected, therefore, that future research will involve less guesswork and will be conducted more efficiently than it was during the past VTV project. Some supply problems, which resulted in lengthy delays in obtaining certain precursor chemicals, have also been resolved, so that more material should be available for use in research on the polymers.

Another significant positive factor is simply the higher level of accumulated knowledge about the making of SP films which is available now than existed two years ago.

On the other hand, it is still difficult to project the time and cost necessary to solve the outstanding problems, with a good confidence level, without a clear understanding of the causes of the problems. At this point in time there are only hypotheses, or speculations about the root

causes, without clear supporting evidence.

For example, one reason for the loss of contrast with time might be an incompatibility between the fluorination in the suspending polymer and the nitrocellulose that coats the crystals. However, this has not been established. The purpose of the fluorination is to match refractive indices. Work is currently underway at RFI to achieve refractive index matching by other means, thereby eliminating the need for fluorination. It is too early to tell, however, if this will solve the loss-of-contrast problem.

As another example, the crazing problem appears to be related to mechanical stresses imposed on the film by the rigid substrates, which expand and contract at different rates than the film, and which shear the film when the cell is subject to bending forces. It is not clear, however, if mechanical stress is really the root cause. If it is, then a lower viscosity material (prior to cure) might help to alleviate the problem because less time would be required for the material to flow through the cell, and for the plates to relax prior to the cure cycle. Otherwise, no other working hypotheses to explain the crazing exist at this time.

The cause of the remaining major problem - the blue colour - is well understood, and the general nature of the solution is known. However, in this case, it is clear that a great deal of work will be necessary to develop the new types of crystals required to change the colour, and that this will not be feasible in the short term. Therefore, it is not anticipated that a colour-neutral SP film will be developed within a few years.

From this perspective, a period of 2-3 years with a manpower of 2-3 people per year to solve the contrast and crazing problems seems reasonable, but cannot be claimed to be free of risk.

To improve the degassing of the liquid crystal fluid and polymer mixture, a proper mixing setup and processing environment would be required. After the equipment has been setup, another six months may be required to perfect the process. More studies are also required to improve and evaluate the uniformity of the NCAP films which are spin-coated directly onto the visor. This may require an additional six months. The deposition of ITO on NCAP films is another area which may require six months of development time. All of this development work can be done within LSL.

The other area of technological improvement, namely the reduction of the liquid crystal droplet sizes and the tailoring of the indices of refraction of the liquid crystal fluids, will definitely require partnership arrangements with liquid crystal manufacturers and with other chemical manufacturers. That work may take years to complete. On the other hand, the interest which liquid crystal companies might take in such a venture would certainly depend on the nature of their businesses. Unfortunately, except for the VTV application, there does not appear to be a market for liquid crystal fluids having almost zero birefringence. This, however, is a requirement for the VTV since fluids with only low birefringence (~ 0.07) still exhibit unacceptable haze for this application, and cannot be used.

In light of these technical difficulties, a major portion of which is beyond Litton System's own R&D capabilities, it appears unlikely that LSL would be able to develop a compliant VTV in the near future.

Perhaps the best scenario is to wait for some period of time and watch the developments in suspended particle technology. When an appropriate breakthrough in the technology occurs, then the possibility of continuing the development of the VTV could be considered.